Supporting Information for

Merging organocatalysis with transition metal catalysis and using O_2 as the oxidant for enantioselective C-H functionalization of aldehydes

Yong-Long Zhao, Yao Wang, Xiu-Qin Hu and Peng-Fei Xu*

State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering,Lanzhou University, Lanzhou 730000, P. R. China
Fax: 86-931-8915557 E-mail: xupf@lzu.edu.cn

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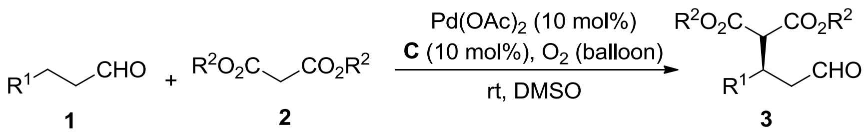
1. General Information

Chemicals and solvents were either purchased from commercial suppliers or purified by standard procedures as specified in *Purification of Laboratory Chemicals*, 4th Ed (Armarego, W. L. F.; Perrin, D. D. Butterworth Heinemann: 1997). Analytical thin-layer chromatography (TLC) was performed on silica gel plates with F-254 indicator and compounds were visualized by irradiation with UV light and/or by treatment with a solution of phosphomolybdic acid in ethanol followed by heating or $KMnO_4$ stain. Flash chromatography was carried out utilizing silica gel (200-300 mesh). ^{1}H NMR, ^{13}C NMR spectra were recorded on a Bruker AM-400 spectrometer (400 MHz 1H, 100 MHz ^{13}C). The spectra were recorded in $CDCl_3$ as the solvent at room temperature, ^{1}H and ^{13}CNMR chemical shifts are reported in ppm relative to either the residual solvent peak (^{13}C) (δ = 77.00 ppm) or TMS (^{1}H) (δ = 0 ppm) as an internal standard. Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, dd = doublet), integration，coupling constant (Hz) and assignment. Data for ^{13}C NMR are reported as chemical shift. IR spectra were recorded using a Nicolet NEXUS 670 FT-IR instrument and are reported in wavenumbers (cm^{-1}). HRMS were performed on a Bruker Apex II mass instrument (ESI). Enantiomeric excess values were determined by HPLC using a Daicel Chirapak AD-H columb on Water 600/2996 and eluting with *i*-PrOH and *n*-hexane. Optical rotation was measured on the Perkin Elmer 341 polarimeter with $[\alpha]_D$ values reported in degrees; concentration (c) is in g/100 mL.
$Pd(OAc)_2$ and Phenylpropionaldehyde were purchased from *Energy Chemical* (China).

2. Preparation of Substrates

Substrates **1** were prepared by following the procedures in references 1 and 2.

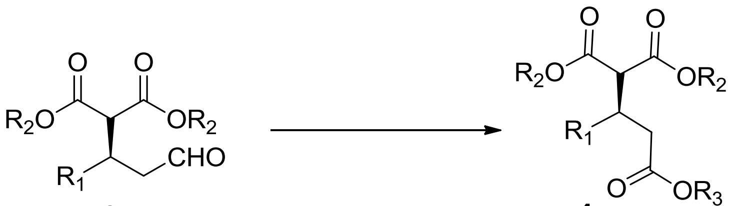
3. General Procedure and Spectral Data of Products[3]



3.1 General procedure for catalytic enantioselective Saegusa oxidation/Michael cascade reaction of malonates 2 to aldehydes 1

$Pd(OAc)_2$ (4.5mg, 0.04 mmol, 10 mol%)， catalyst **C** (6.5 mg, 0.04 mmol, 10 mol%) and dry DMSO (0.5 ml) were added to a dry reaction tube. The tube was then charged with O_2 (using a balloon), and the reaction mixture was stirred at room temperature for 10-20 minutes. Aldehydes **1** (0.4 mmol) and freshly distilled malonates **2** (0.2 mmol) were added subsequently to the above reaction mixture under stirring. After 28-32 h, the reaction was complete (as judged by TLC analysis). The reaction mixture was directly purified by flash column chromatography (eluted with EtOAc/petroleum ether: 1/20 to 1/8) to afford the products **3**.

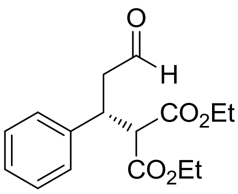
3.2 General procedure for oxidation of aldehydes 3 to carboxylic esters 4



3 **4**

Aldehydes **3** (0.10 mmol) were diluted with 3.0 mL *t*-BuOH and 3.0 mL 1 M NaH_2PO_4 (aq.). 3.0 mL 1 M $KMnO_4$ was added subsequently. After 5 min of vigorous stirring, 5.0 mL saturated $NaHSO_3$ was added and the pH was adjusted to approximately 3 with 1 M HCl. The resulting mixture was extracted 3 times with 10 mL EtOAc, and the combined organic layers were washed with 10 mL of water and 10 mL of brine, and then dried over $MgSO_4$. The organic layer was concentrated in vacuum and the residual acid was dissolved in 2 ml EtOH or MeOH. $SOCl_2$ (2.0 mmol) was added dropwise at 0 ^{o}C. The solution was stirred overnight at room temperature and then quenched with saturated Na_2CO_3. The resulting mixture was extracted 3 times with 10 mL EtOAc, and the combined organic layers were washed with water and brine, and then dried over Na_2SO_4. The organic layer was concentrated in vacuum. The crude product was subjected to FC on silica gel (EtOAc/ petroleum ether: 1/15 to 1/10) to give corresponding carboxylic esters **4**.

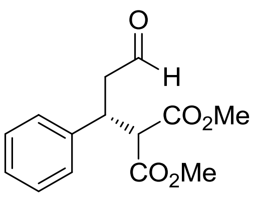
3.3 Analytical data of chiral aldehydes 3



3a

(*R*)-2-(3-Oxo-1-phenylpropyl)malonic acid diethyl ester (3a).

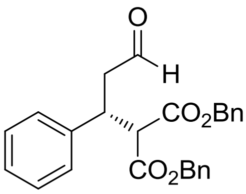
Colourless liquid; Yield: 64%; IR (KBr): 3435, 2983, 2938, 1749, 1728, 1450, 1452, 1370, 1310, 1250, 1175, 1030, 863, 766, 702 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.60 (t, *J* = 1.6 Hz, 1H), 7.32-7.15 (m, 5H), 4.21 (q, *J* = 7.2 Hz, 2H), 4.02 (td, *J* = 9.6 Hz, *J* = 5.2 Hz, 1H), 3.95 (q, *J* = 7.2 Hz, 2H), 3.71 (d, *J* = 10.0 Hz, 1H), 3.00-2.81 (m, 2H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.00 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.0, 168.0, 167.4, 139.8, 128.7, 128.5, 128.4, 128.1, 127.5, 61,8, 61.4, 57.5, 47.4, 39.6, 14.0, 13.7; The product was converted to corresponding ester **4a**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90∶10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 9.03 min (major), t_R = 14.03 min (minor), 95% ee; $[\alpha]_D^{20}$ = –33 (*c* 0.66, $CHCl_3$); HRMS (ESI): calculated $[M+H]^+$ for $C_{16}H_{21}O_5$: 293.1384, found $[M+H]^+$: 293.1379.



3b

(*R*)-2-(3-Oxo-1-phenylpropyl)malonic acid dimethyl ester (3b).

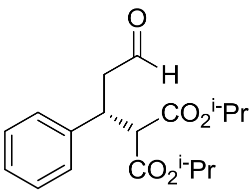
Colourless liquid; Yield: 59%; IR (KBr): 3431, 2955, 1734, 1496, 1452, 1319, 1283, 1253, 1157, 1022, 754, 702 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.60 (t, *J* = 1.6 Hz, 1H), 7.32-7.20 (m, 5H), 4.03 (td, *J* = 9.2 Hz, *J* = 5.2 Hz, 1H), 3.75 (d, *J* = 9.6 Hz, 1H), 3.74 (s, 3H), 3.50 (s, 3H), 2.99-2.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.9, 168.4, 167.8, 139.7, 128.8, 128.0, 127.6, 57.3, 52.7, 52.5, 47.2, 39.5; The product was converted to corresponding ester **4b**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 211.0 nm), t_R = 11.38 min (major), t_R = 13.80 min (minor), 94% ee; $[\alpha]_D^{20}$ = –29 (*c* 0.63, $CHCl_3$); HRMS (ESI): calculated $[M+H]^+$ for $C_{14}H_{17}O_5$: 265.1071, found $[M+H]^+$: 265.1065.



3C

(*R*)-2-(3-Oxo-1-phenylpropyl)malonic acid dibenzyl ester (3c).

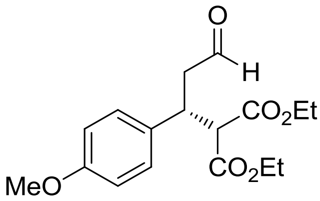
White solid; Yield: 57%; IR (KBr): 3483, 3063, 3033, 1746, 1727, 1496, 1454, 1382, 1315, 1251, 1171, 1153, 1089, 1025, 998, 905, 747, 700, 587 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.55 (t, *J* = 1.6Hz, 1H), 7.36-7.04 (m, 15H), 5.16 (s, 2H), 4.91 (s, 2H), 4.08-4.03 (m, 1H), 3.85 (d, *J* = 10.0 Hz, 1H), 2.88 (dd, *J* = 1.6 Hz, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.8, 167.7, 167.2, 139.6, 135.0, 134.9, 128.8, 128.6, 128.5, 128.4, 128.3 (128.30), 128.3 (128.28), 128.2, 128.1, 127.5, 67.5, 67.2, 57.5, 47.2, 39.5; The product was converted to corresponding ester **4c**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90∶10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 25.59 min (major), t_R = 35.21 min (minor), 89% ee; $[\alpha]_D^{20}$ = –12 (*c* 0.49, $CHCl_3$); HRMS (ESI): calculated $[M+NH_4]^+$ for $C_{26}H_{28}NO_5$: 434.1962, found $[M+NH_4]^+$: 434.1955.



3d

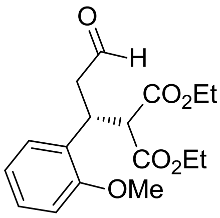
(*R*)-2-(3-Oxo-1-phenylpropyl)malonic acid diisopropyl ester (3d).

Colourless liquid; Yield: 66%; IR (KBr): 3434, 2983, 2936, 1745, 1727, 1456, 1374, 1311, 1283, 1254, 1175, 1104, 908, 760, 702 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.60 (t, *J* = 1.6 Hz, 1H), 7.31-7.18 (m, 5H), 5.10-5.03 (m, 1H), 4.82-4.73 (m, 1H), 3.99 (td, *J* = 9.6 Hz, *J* = 4.8 Hz, 1H), 3.65 (d, *J* = 10.4 Hz, 1H), 2.96-2.80 (m, 2H), 1.25 (d, *J* = 6.4 Hz, 3H), 1.24 (d, *J* = 6.4 Hz, 3H), 1.05 (d, *J* = 6.4 Hz, 3H), 0.95 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.2, 167.6, 167.0, 139.9, 128.6, 128.2, 127.4, 69.5, 69.0, 57.8, 47.7, 39.4, 21.7, 21.5, 21.3 (21.32), 21.3 (21.25); The product was converted to corresponding ester **4d**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 6.90 min (major), t_R = 10.18 min (minor), 94% ee; $[\alpha]_D^{20}$ = –39 (*c* 0.74, $CHCl_3$); HRMS (ESI): calculated $[M+H]^+$ for $C_{18}H_{25}O_5$: 321.1697, found $[M+H]^+$: 321.1694.



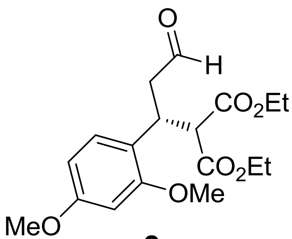
3e

(*R*)-2-(3-Oxo-1-(4-methoxyphenyl)propyl)malonic acid diethyl ester (3e). Colourless liquid; Yield: 72%; IR (KBr): 3311, 2981, 2939, 1746, 1728, 1601, 1495, 1465, 1302, 1247, 1154, 1028, 756 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.57 (t, *J* = 1.6 Hz, 1H), 7.20-7.12 (m, 2H), 6.85-6.75 (m, 2H), 4.19 (q, *J* = 7.2 Hz, 2H), 4.01-3.90 (m, 3H), 3.75 (s, 3H), 3.66 (d, *J* = 10.0 Hz, 1H), 2.93-2.75 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.3, 168.0, 167.5, 158.8, 131.7, 129.3, 129.2, 114.0, 113.8, 61.7, 61.4, 57.7, 55.2, 47.5, 38.8, 14.0, 13.8; The product was converted to corresponding ester **4e**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90∶10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 13.57 min (major), t_R = 26.15 min (minor), 94% ee; $[\alpha]_D^{20}$ = –29 (*c* 1.19, $CHCl_3$); HRMS (ESI): calculated $[M+Na]^+$ for $C_{17}H_{22}NaO_6$: 345.1309, found $[M+Na]^+$: 345.1304.



3f

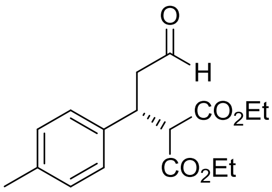
(*R*)-2-(3-Oxo-1-(2-methoxyphenyl)propyl)malonic acid diethyl ester (3f). Colourless liquid; Yield: 51%; IR (KBr): 3334, 2981, 2940, 1747, 1728, 1601, 1495, 1465, 1369, 1302, 1247, 1176, 1154, 1028, 861, 756 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.60 (t, *J* = 1.6 Hz, 1H), 7.24-7.15 (m, 2H), 6.89-6.83 (m, 2H), 4.24-4.15 (m, 3H), 4.06 (d, *J* = 10.0 Hz, 1H), 3.92 (q, *J* = 7.2 Hz, 2H), 3.85 (s, 3H), 3.02-2.94 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H), 0.99 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.1, 168.4, 167.8, 157.4, 130.2, 128.7, 127.3, 120.6, 110.9, 61.6, 61.2, 55.3, 55.0, 45.8, 36.3, 14.0, 13.7; The product was converted to corresponding ester **4f**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 97:3, flow rate 1 mL/min, λ = 210.5 nm), t_R = 21.10 min (major), t_R = 24.34 min (minor), 95% ee; $[\alpha]_D^{20}$ = –34 (*c* 0.99, $CHCl_3$); HRMS (ESI): calculated $[M+Na]^+$ for $C_{17}H_{22}NaO_6$: 345.1309, found $[M+Na]^+$: 345.1305.



3g

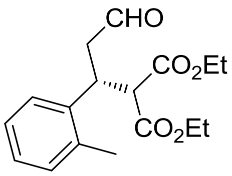
(*R*)-2-(3-Oxo-1-(2, 4-dimethoxyphenyl)propyl)malonic acid diethyl ester (3g). Yellow liquid; Yield: 53%; IR (KBr): 3021, 2984, 2933, 1726, 1613, 1588, 1507, 1464, 1296, 1215, 1159, 1134, 1034, 928, 836, 757, 669 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.58 (t, *J*

= 1.6 Hz, 1H), 7.06 (d, *J* = 8.4 Hz, 1H), 6.42-6.35 (m, 2H), 4.22-4.08 (m, 3H), 4.02 (d, *J* = 10.4 Hz, 1H), 3.94 (q, *J* = 7.2 Hz, 2H), 3.82 (s, 3H), 3.76 (s, 3H), 2.99-2.75 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H), 1.02 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.3, 168.5, 167.9, 160.3, 158.4, 130.8, 119.6, 104.1, 98.9, 61.5, 61.2, 55.3 (55.29), 55.3 (55.27), 55.2, 45.9, 35.9, 14.0, 13.8; The product was converted to corresponding ester **4g**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 13.92 min (major), t_R = 17.70 min (minor), 87% ee; $[\alpha]_D^{20}$ = –32 (*c* 1.56, $CHCl_3$); HRMS (ESI): calculated $[M+NH_4]^+$ for $C_{18}H_{28}NO_7$: 370.1860, found $[M+NH_4]^+$: 370.1865.



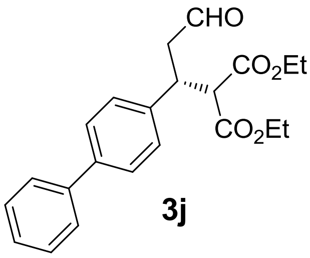
3h

(*R*)-2-(3-Oxo-1-(4-methylphenyl)propyl)malonic acid diethyl ester (3h). Yellow oil; Yield: 64%; IR (KBr): 3428, 2982, 2924, 1728, 1514, 1449, 1369, 1308, 1247, 1171, 1153, 1028, 816 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.59 (t, *J* = 1.6 Hz, 1H), 7.12 (q, *J* = 8.0 Hz, 4H), 4.21 (q, *J* = 7.2 Hz, 2H), 4.02-3.90 (m, 3H), 3.69 (d, *J* = 10.4 Hz, 1H), 2.95-2.80 (m, 2H), 2.30 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.04 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.3, 168.1, 167.5, 137.1, 136.7, 129.4, 127.9, 61.7, 61.4, 57.6, 47.5, 39.2, 21.0, 14.0, 13.8; The product was converted to corresponding ester **4h**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 9.50 min (major), t_R = 14.68 min (minor), >99% ee; $[\alpha]_D^{20}$ = –24 (*c* 0.72, $CHCl_3$); HRMS (ESI): calculated $[M+Na]^+$ for $C_{17}H_{22}NaO_5$: 329.1359, found $[M+Na]^+$: 329.1355.



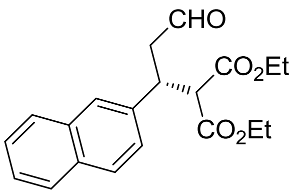
3i

(*R*)-2-(3-Oxo-1-(2-methylphenyl)propyl)malonic acid diethyl ester (3i). Yellow oil; Yield: 61%; IR (KBr): 3365, 2981, 2937, 1748, 1728, 1494, 1465, 1447, 1369, 1305, 1252, 1177, 1153, 1150, 1031, 760, 729 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.57 (s, 1H), 7.15-7.07 (m, 4H), 4.29 (td, *J* = 9.6 Hz, *J* = 4.8 Hz, 1H), 4.21 (q, *J* = 7.2 Hz, 2H), 3.96-3.88 (m, 2H), 3.73 (d, *J* = 10.4 Hz, 1H), 2.97-2.83 (m, 2H), 2.47 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 0.97 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.0, 168.2, 167.5, 138.3, 136.5, 130.8, 127.1, 126.4, 126.3, 61.8, 61.4, 57.1, 48.1, 34.3, 19.8, 14.0, 13.6; The product was converted to corresponding ester **4i**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 6.43 min (major), t_R = 9.42 min (minor), 94% ee; $[\alpha]_D^{20}$ = –16 (*c* 1.05, $CHCl_3$); HRMS (ESI): calculated $[M+Na]^+$ for $C_{17}H_{22}NaO_5$: 329.1359, found $[M+Na]^+$: 329.1356.



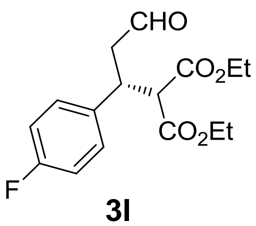
3j

(*R*)-2-(1-(Biphenyl-4-yl)-3-oxopropyl)malonic acid diethyl ester (3j). Yellow oil; Yield: 65%; IR (KBr): 3514, 3442, 3029, 2982, 2938, 1747, 1728, 1487, 1447, 1369, 1314, 1300, 1250, 1176, 1156, 1096, 1030, 1009, 859, 842, 764, 735, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.65 (t, *J* = 1.6 Hz, 1H), 7.59-7.25 (m, 9H), 4.23 (q, *J* = 7.2 Hz, 2H), 4.16-4.05 (m, 1H), 3.99 (q, *J* = 7.2 Hz, 2H), 3.77 (d, *J* = 10.0 Hz, 1H), 3.03-2.89 (m, 2H), 1.29 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.0, 167.9, 167.4, 140.5, 140.3, 138.8, 128.7, 128.5, 127.3, 126.9, 61.7, 61.4, 57.4, 47.4, 39.1, 14.0, 13.7; The product was converted to corresponding ester **4j**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 254.0 nm), t_R = 13.72 min (major), t_R = 30.09 min (minor), 95% ee; $[\alpha]_D^{20}$ = –21 (*c* 1.91, $CHCl_3$); HRMS (ESI): calculated $[M+NH_4]^+$ for $C_{22}H_{28}NO_5$: 386.1962, found $[M+NH_4]^+$: 386.1957.



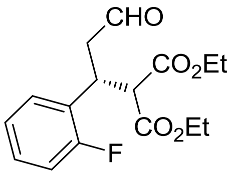
3k

(*R*)-2-(1-(naphthalen-2-yl)-3-oxopropyl)malonic acid diethyl ester (3k). Colourless liquid; Yield: 47%; IR (KBr): 3356, 2982, 2937, 1747,1750, 1446, 1369, 1300, 1249, 1177, 1154, 1029, 859, 820, 750, 650 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.63 (t, *J* = 1.6 Hz, 1H), 7.82-7.38 (m, 7H), 4.27-4.18 (m, 3H), 3.96-3.87 (m, 2H), 3.85 (d, *J* = 10.0 Hz, 1H), 3.04-3.00 (m, 2H), 1.28 (t, *J* = 7.2 Hz, 3H), 0.94 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.0, 168.0, 167.4, 137.3, 133.3, 132.7, 128.5, 127.8, 127.6, 127.2, 126.3, 126.0, 125.9, 61.8, 61.5, 57.5, 47.4, 39.6, 14.0, 13.7; The product was converted to corresponding ester **4k**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 14.72 min (major), t_R = 24.56 min (minor), 94% ee; $[\alpha]_D^{20}$ = –23 (*c* 1.33, $CHCl_3$); HRMS (ESI): calculated $[M+Na]^+$ for $C_{20}H_{22}NaO_5$: 365.1359, found $[M+Na]^+$: 365.1355.



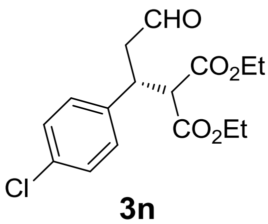
3l

(*R*)-2-(3-Oxo-1-(4-fluorophenyl)propyl)malonic acid diethyl ester (3l). White solid; Yield: 63%; IR (KBr): 3429, 2984, 2938, 2908, 1748, 1728, 1605, 1511, 1466, 1370, 1306, 1279, 1250, 1226, 1177, 1161, 1100, 1031, 840 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.60 (s, 1H), 7.23 (dd, *J* = 8.4 Hz, *J* = 5.2 Hz, 2H), 6.98 (t, *J* = 8.8 Hz, 2H), 4.21 (q, *J* = 7.2 Hz, 2H)，4.05-3.92 (m, 3H), 3.67 (d, *J* = 10.0 Hz, 1H), 2.98-2.82 (m, 2H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.04 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.7, 167.8, 167.3, 163.1, 160.7, 135.6, 135.5, 129.8, 129.7, 115.6, 115.4, 61.8, 61.5, 57.4, 47.5, 38.6, 14.0, 13.7; The product was converted to corresponding ester **4l**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 10.26 min (major), t_R = 19.45 min (minor), 94% ee; $[\alpha]_D^{20}$ = –33 (*c* 1.13, $CHCl_3$); HRMS (ESI): calculated $[M+NH_4]^+$ for $C_{16}H_{23}NFO_5$: 328.1555, found $[M+NH_4]^+$: 328.1559.



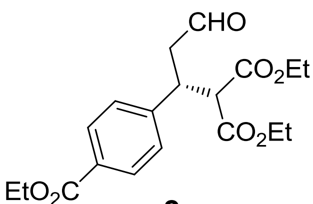
3m

(*R*)-2-(3-Oxo-1-(2-fluorophenyl)propyl)malonic acid diethyl ester (3l). Colourless liquid; Yield: 55%; IR (KBr): 3432, 2983, 2933, 1748, 1730, 1585, 1493, 1456, 1370, 1310, 1251, 1233, 1177, 1154, 1109, 1030, 860, 761 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.63 (s, 1H), 7.29-7.18 (m, 2H), 7.09-6.98 (m, 2H), 4.24-4.16 (m, 3H), 3.95 (q, *J* = 7.2 Hz, 2H), 3.89 (d, *J* = 10.4, 1H), 2.98-2.95 (m, 2H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.01 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.7, 167.9, 167.4, 162.2, 159.7, 130.6, 130.5, 129.3, 129.2, 126.7, 126.6, 124.3, 124.2, 116.0, 115.8, 61.8, 61.5, 55.6, 46.3, 34.7, 14.0, 13.7; The product was converted to corresponding ester **4m**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 9.75 min (major), t_R = 14.08 min (minor), 96% ee; $[\alpha]_D^{20}$ = –29 (*c* 1.32, $CHCl_3$); HRMS (ESI): calculated $[M+NH_4]^+$ for $C_{16}H_{23}NFO_5$: 328.1555, found $[M+NH_4]^+$: 328.1552.



3n

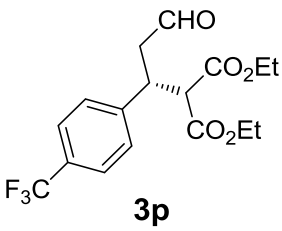
(*R*)-2-(3-Oxo-1-(4-chlorophenyl)propyl)malonic acid diethyl ester (3n). Colourless liquid; Yield: 53%; IR (KBr): 3435, 2983, 2939, 1749, 1730, 1493, 1466, 1414, 1391, 1370, 1308, 1250, 1176, 1157, 1111, 1094, 1031, 1015, 862, 832, 733, 539 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 9.60 (s, 1H), 7.26 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.8 Hz, 2H), 4.20 (q, *J* = 7.2 Hz, 2H), 4.04-3.94 (m, 3H), 3.68 (d, *J* = 9.6 Hz, 1H), 2.99-2.83 (m, 2H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.4, 167.7, 167.2, 138.4, 133.2, 129.5, 128.8, 61.8, 61.5, 57.2, 47.3, 38.7, 14.0, 13.7; The product was converted to corresponding ester **4n**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 11.02 min (major), t_R = 19.59 min (minor), 95% ee; $[\alpha]_D^{20}$ = –29 (*c* 1.44, $CHCl_3$); HRMS (ESI): calculated $[M+Na]^+$ for $C_{16}H_{19}NaClO_5$: 349.0813, found $[M+Na]^+$: 349.0817.



3o

(*R*)-2-(3-Oxo-1-(4-ethoxycarbonylphenyl)propyl)malonic acid

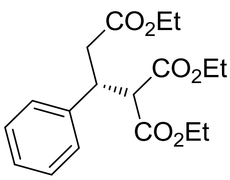
diethyl ester (3o). Colourless liquid; Yield: 45%; IR (KBr): 3425, 2983, 2938, 1750, 1723, 1611, 1576, 1466, 1447, 1278, 1252, 1178, 1157, 1107, 1021, 858, 775, 708 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.61 (s, 1H), 7.97 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 4.35 (q, *J* = 7.2 Hz, 2H), 4.22 (q, *J* = 7.2 Hz, 2H), 4.19-4.05 (m, 1H), 3.99-3.92 (m, 2H), 3.74 (d, *J* = 10.0 Hz, 1H), 3.03-2.88 (m, 2H), 1.38 (t, *J* = 7.2 Hz, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.3, 167.7, 167.2, 166.2, 145.2, 129.9, 129.7, 128.2, 61.9, 61.6, 61.0, 57.0, 47.3, 39.2, 14.3, 14.0, 13.8; The product was converted to corresponding ester **4o**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 230.0 nm), t_R = 22.04 min (major), t_R = 40.82 min (minor), 94% ee; $[\alpha]_D^{20}$ = –22 (*c* 1.07, $CHCl_3$); HRMS (ESI): calculated $[M+NH_4]^+$ for $C_{19}H_{28}NO_7$: 382.1860, found $[M+NH_4]^+$: 382.1864.



3p

(*R*)-2-(3-Oxo-1-(4-trifluoromethylphenyl)propyl)malonic acid diethyl ester (3p). Colourless liquid; Yield: 47%; IR (KBr): 3503, 3023, 2985, 1747, 1729, 1620, 1422, 1371, 1327, 1252, 1217, 1167, 1128, 1069, 1031, 1019, 844, 758, 668, 608 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.62 (s, 1H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 2H), 4.21 (q, *J* = 7.2 Hz, 2H), 4.10 (td, *J* = 9.6 Hz, *J* = 4.8 Hz, 1H), 3.97 (q, *J* = 7.2 Hz, 2H), 3.73 (d, *J* = 10.0 Hz, 1H), 3.05-2.90 (m, 2H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.02 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.1, 167.7, 167.2, 144.2, 129.9, 129.5, 128.8, 128.6, 125.6 (125.61), 125.6 (125.57), 125.5 (125.53), 125.5 (125.50), 125.3, 122.6, 61.9, 61.6, 56.9, 47.2, 39.0, 14.0, 13.7; The product was converted to corresponding ester **4p**. The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90： 10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 9.77 min (major), t_R = 16.30 min (minor), 94% ee; $[\alpha]_D^{20}$ = –17 (*c* 2.06, $CHCl_3$); HRMS (ESI): calculated $[M+NH_4]^+$ for $C_{17}H_{23}NF_3O_5$: 378.1523, found $[M+NH_4]^+$: 378.1528.

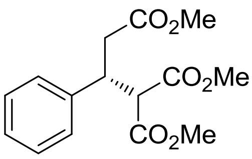
3.4 Analytical data of derivatization products 4



4a

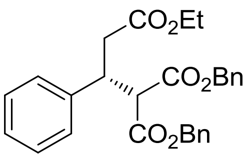
(*R*)-2-Ethyloxycarbonyl-3-phenylpetanedioic acid 1,5-diethyl ester

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.15 (m, 5H), 4.16 (q, *J* = 7.2 Hz, 2H)， 4.10-3.85 (m, 5H), 3.73 (d, *J* = 10.4 Hz, 1H), 2.90-2.65 (m, 2H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.08 (t, *J* = 7.2 Hz, 3H), 0.99 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 168.0, 167.5, 139.8, 128.3, 128.2, 127.3, 61.6, 61.3, 60.4, 57.4, 41.5, 38.8, 14.0, 14.0 (13.97), 13.7; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 9.03 min (major), t_R = 14.03 min (minor), 95% ee;



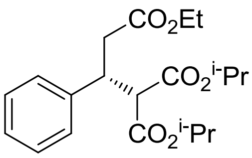
4b

(*R*)-2-Methyloxycarbonyl-3-phenylpetanedioic acid 1,5-dimethyl ester (4b). [1]H NMR (400 MHz, $CDCl_3$): δ 7.32-7.18 (m, 5H), 3.93 (td, *J* = 9.6 Hz, *J* = 4.8 Hz, 1H), 3.79 (d, *J* = 10.0 Hz, 1H), 3.75 (s, 3H), 3.54 (s, 3H), 3.48 (s, 3H), 2.89-2.72 (m, 2H); [13]C NMR (100 MHz, $CDCl_3$): δ 171.5, 168.4, 167.9, 139.8, 128.5, 127.9, 127.4, 57.0, 52.7, 52.4, 51.6, 41.4, 38.3; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 211.0 nm), t_R = 11.38 min (major), t_R = 13.80 min (minor), 94% ee.



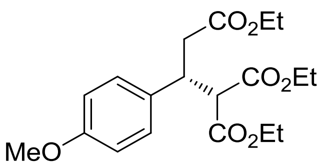
4C

(*R*)-2-Ethyloxycarbonyl-3-phenylpetanedioic acid 1,5-dibenzyl ester (4c). [1]H NMR (400 MHz, $CDCl_3$): δ 7.34-7.01 (m, 15H), 5.16 (s, 2H), 4.87 (s, 2H), 3.99-3.91 (m, 3H), 3.90 (d, *J* = 12.0 Hz, 1H)， 2.84-2.64 (m, 2H), 1.06 (t, *J* = 7.2 Hz, 3H); [13]C NMR (100 MHz, $CDCl_3$): δ 171.0, 167.7, 167.3, 139.6, 135.1, 135.0, 128.6, 128.5, 128.4 (128.42), 128.4 (128.40), 128.2, 128.1 (128.13), 128.1 (128.12), 127.4, 67.3, 67.1, 60.4, 57.3, 41.5, 38.6, 14.0; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 25.59 min (major), t_R = 35.207 min (minor), 89% ee.



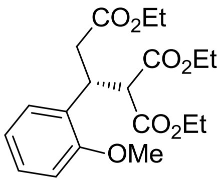
4d

(*R*)-2-Ethyloxycarbonyl-3-phenylpetanedioic acid 1,5-diisopropyl ester (4d). [1]H NMR (400 MHz, $CDCl_3$): δ 7.30-7.15 (m, 5H), 5.14-5.01 (m,1H), 4.83-4.71 (m, 1H), 4.02-3.91 (m, 2H), 3.89 (td, *J* = 10.4 Hz, *J* = 4.4 Hz, 1H), 3.68 (d, *J* = 10.4 Hz, 1H), 2.89-2.65 (m, 2H), 1.28-1.23 (m, 6H), 1.08 (t, *J* = 7.2 Hz, 3H), 1.03 (d, *J* = 6.4 Hz, 3H), 0.94 (d, *J* = 6.4 Hz, 3H); [13]C NMR (100 MHz, $CDCl_3$): δ 171.1, 167.6, 167.0, 139.9, 128.3 (128.32), 128.3 (128.27), 127.2, 69.3, 68.8, 60.3, 57.6, 41.4, 39.0, 21.7, 21.5, 21.3, 21.2, 14.0; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 6.90 min (major), t_R = 10.18 min (minor), 94% ee.



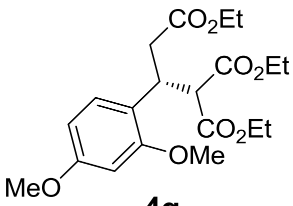
4e

(*R*)-2-Ethyloxycarbonyl-3-(4-methoxyphenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (4e). [1]H NMR (400 MHz, $CDCl_3$): δ 7.16 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.8 Hz, 2H), 4.21 (q, *J* = 7.2 Hz, 2H), 4.24-3.91 (m, 4H), 3.87 (td, *J* = 10.4 Hz, *J* = 4.4 Hz, 1H), 3.76 (s, 3H), 3.68 (d, *J* = 10.0 Hz, 1H), 2.85-2.62 (m, 2H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.10 (t, *J* = 7.2 Hz, 3H), 1.02 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 168.1, 167.6, 158.7, 131.8, 129.2, 113.7, 61.6, 61.3, 60.3, 57.6, 57.1, 40.8, 38.9, 14.0, 13.8; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90∶10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 13.57 min (major), t_R = 26.15 min (minor), 94% ee.



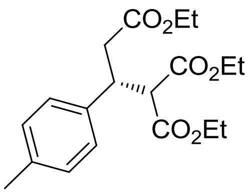
4f

(*R*)-2-Ethyloxycarbonyl-3-(2-methoxyphenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (4f). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (t, *J* = 8.0 Hz, 2H), 6.84 (t, *J* = 6.8 Hz, 2H), 4.20 (qd, *J* = 7.2 Hz, *J* = 1.2 Hz, 2H), 4.12 (d, *J* = 10.4 Hz, 1H), 4.06 (td, *J* = 10.0 Hz, *J* = 4.0 Hz, 1H), 4.00-3.87 (m, 4H), 3.85 (s, 3H), 2.98 (dd, *J* = 15.6 Hz, *J* = 9.6 Hz, 1H), 2.79 (dd, *J* = 15.6 Hz, *J* = 4.4 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.09 (t, *J* = 7.2 Hz, 3H), 0.98 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.7, 168.5, 168.0, 157.7, 130.9, 128.5, 127.3, 120.3, 110.8, 61.4, 61.0, 60.2, 55.3, 54.8, 38.9, 36.5, 14.0, 13.7; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 97:3, flow rate 1 mL/min, λ = 210.5 nm), t_R = 21.10 min (major), t_R = 24.34 min (minor), 95% ee.



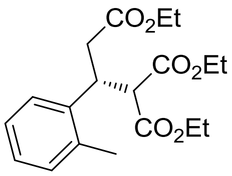
4g

(*R*)-2-Ethyloxycarbonyl-3-(2,4-dimethoxyphenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (4g). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.06 (d, *J* = 8.4 Hz, 1H), 6.42-6.32 (m, 2H), 4.23-4.15 (m, 2H), 4.08 (d, *J* = 10.4 Hz, 1H), 4.02-3.88 (m, 5H), 3.82 (s, 3H), 3.76 (s, 3H), 2.94 (dd, *J* = 15.6 Hz, *J* = 10.4 Hz, 1H), 2.74 (dd, *J* = 15.2 Hz, *J* = 4.0 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.11 (t, *J* = 7.2 Hz, 3H), 1.01 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 168.6, 168.1, 160.1, 158.6, 131.4, 119.6, 103.8, 98.8, 61.4, 61.0, 60.1, 55.3, 55.2, 55.0, 38.4, 36.7, 14.1, 13.8; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 13.92 min (major), t_R = 17.70 min (minor), 87% ee.



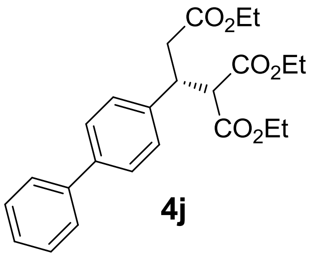
4h

(*R*)-2-Ethyloxycarbonyl-3-(4-methylphenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (4h). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.10 (dd, *J* = 8.0 Hz, *J* = 24.0 Hz, 4H), 4.21 (q, *J* = 7.2 Hz, 2H), 4.01-3.83 (m, 5H), 3.71 (d, *J* = 10.4 Hz, 1H), 2.82 (dd, *J* = 15.6 Hz, *J* = 4.8 Hz, 1H), 2.70 (dd, *J* = 15.6 Hz, *J* = 10.0 Hz, 1H), 2.28 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.10 (t, *J* = 7.2 Hz, 3H), 1.01 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 168.1, 167.5, 136.7, 129.0, 128.0, 61.6, 61.2, 60.3, 57.4, 41.1, 38.8, 21.0, 14.0 (13.98), 14.0 (13.96), 13.7; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 9.50 min (major), t_R = 14.68 min (minor), >99% ee.



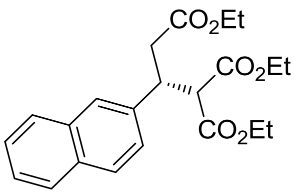
4i

(*R*)-2-Ethyloxycarbonyl-3-(2-methylphenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (4i). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18-7.05 (m, 4H), 4.27-4.20 (m, 3H), 3.99-3.87 (m, 4H), 3.74 (d, *J* = 10.4 Hz, 1H), 2.85 (dd, *J* = 15.6 Hz, *J* = 4.8 Hz, 1H), 2.73 (dd, *J* = 15.6 Hz, *J* = 10.0 Hz, 1H), 2.47 (s, 3H), 1.29 (t, *J* = 7.2 Hz, 3H), 1.08 (t, *J* = 7.2 Hz, 3H), 0.96 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 168.2, 167.6, 138.3, 137.0, 130.6, 126.9, 126.4, 126.0, 61.7, 61.3, 60.4, 57.3, 39.0, 36.1, 19.7, 14.1, 13.9, 13.6; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 6.43 min (major), t_R = 9.42 min (minor), 94% ee.



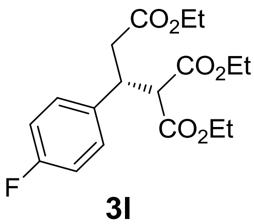
4j

(*R*)-1,1-diethyl 3-ethyl 2-(biphenyl-4-yl)propane-1,1,3-tricarboxylate (4j). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58-7.25 (m, 9H), 4.23 (q, *J* = 7.2 Hz, 2H), 4.04-3.92 (m, 5H), 3.77 (d, *J* = 10.4 Hz, 1H), 2.88 (dd, *J* = 15.6 Hz, *J* = 4.4 Hz, 1H), 2.77 (dd, *J* = 15.6 Hz, *J* = 10.4 Hz, 1H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.10 (t, *J* = 7.2 Hz, 3H), 1.00 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 168.0, 167.5, 140.7, 140.0, 138.9, 128.7, 128.6, 127.2, 127.0, 126.9, 61.7, 61.4, 60.4, 57.3, 41.2, 38.7, 14.0 (14.03), 14.0 (14.00), 13.7; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 254.0 nm), t_R = 13.72 min (major), t_R = 30.09 min (minor), 95% ee.



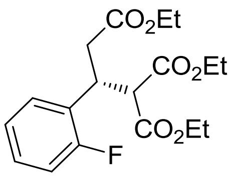
4k

(*R*)-1,1-diethyl 3-ethyl 2-(naphthalen-2-yl)propane-1,1,3-tricarboxylate (4k). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79-7.70 (m, 4H), 7.45-7.35 (m, 3H), 4.23 (q, *J* = 7.2 Hz, 2H), 4.12 (td, *J* = 10.0 Hz, *J* = 4.8 Hz, 1H), 3.99-3.82 (m, 5H), 2.96-2.80 (m, 2H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.04 (t, *J* = 7.2 Hz, 3H), 0.91 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 168.0, 167.5, 137.4, 133.2, 132.6, 128.1, 127.8, 127.5, 127.2, 126.2, 126.0, 125.8, 61.7, 61.3, 60.4, 57.3, 41.5, 38.7, 14.0 (14.03), 14.0 (13.97), 13.7; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90:10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 14.72 min (major), t_R = 24.56 min (minor), 94% ee.



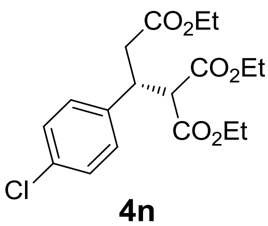
3l

(*R*)-2-Ethyloxycarbonyl-3-(4-fluorophenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (3l). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (dd, *J* = 8.8 Hz, *J* = 5.2 Hz, 2H) ,6.96 (t, *J* = 8.8 Hz, 2H), 4.21 (q, *J* = 7.2 Hz, 2H), 4.20-3.87 (m, 5H), 3.69 (d, *J* = 10.4 Hz, 1H), 2.84 (dd, *J* = 15.6 Hz, *J* = 4.8 Hz, 1H), 2.69 (dd, *J* = 15.6 Hz, *J* = 10.4 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.09 (t, *J* = 7.2 Hz, 3H), 1.02 (t, *J* = 7.2 Hz, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.9, 167.8, 167.4, 163.1, 160.7, 135.6, 135.5, 129.9, 129.8, 115.3, 115.1, 61.7, 61.4, 60.4, 57.3, 40.8, 38.8, 14.0 (13.99), 14.0 (13.98), 13.7; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 10.26 min (major), t_R = 19.95 min (minor), 94% ee.



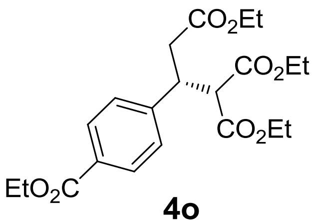
4m

(*R*)-2-Ethyloxycarbonyl-3-(2-fluorophenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (4m). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27-7.15 (m, 3H), 6.96 (dd, *J* = 10.0 Hz, *J* = 8.8 Hz, 1H), 4.23 (q, *J* = 7.2 Hz, 2H), 4.12-3.96 (m, 5H), 3.85 (d, *J* = 10.4 Hz, 1H), 2.88-2.75 (m, 2H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.13 (t, *J* = 7.2 Hz, 3H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.7, 167.6, 167.2, 160.9, 158.4, 130.6, 130.5, 129.0 (129.00), 129.0 (128.96), 128.9, 128.7, 128.6, 117.2, 116.9, 61.9, 61.6, 60.6, 55.3, 37.0, 36.5, 14.0 (14.01), 14.0 (13.98), 13.7; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 9.75 min (major), t_R = 14.08 min (minor), 96% ee.



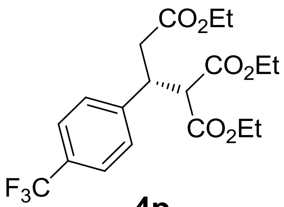
4n

(*R*)-2-Ethyloxycarbonyl-3-(4-chlorophenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (4n). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27-7.16 (m, 4H), 4.21 (q, *J* = 7.2 Hz, 2H), 4.03-3.87 (m, 5H), 3.69 (d, *J* = 10.0 Hz, 1H), 2.83 (dd, *J* = 15.6 Hz, *J* = 4.8 Hz, 1H), 2.69 (dd, *J* = 15.6 Hz, *J* = 10.4 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.11 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.9, 167.8, 167.3, 138.4, 133.1, 129.6, 128.5, 61.8, 61.5, 60.5, 57.1, 40.8, 38.5, 14.0, 13.8; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 11.02 min (major), t_R = 19.59 min (minor), 95% ee.



4o

(*R*)-2-Ethyloxycarbonyl-3-(4-ethoxycarbonylphenyl)pentanedioic acid 1-ethyl ester 5-ethyl ester (4o). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 4.36 (q, *J* = 7.2 Hz, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 4.03-3.92 (m, 5H), 3.75 (d, *J* = 10.4 Hz, 1H), 2.88 (dd, *J* = 15.6 Hz, *J* = 4.4 Hz, 1H), 2.75 (dd, *J* = 15.6 Hz, *J* = 10.0 Hz, 1H), 1.39 (t, *J* = 7.2 Hz, 3H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.10 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8, 167.7, 167.2, 166.3, 145.1, 129.7, 129.6, 128.3, 61.8, 61.5, 60.9, 60.6, 57.0, 41.3, 38.4, 14.3, 14.0, 13.8; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 230.0 nm), t_R = 22.04 min (major), t_R = 40.82 min (minor), 94% ee.



4p

(*R*)-2-Ethyloxycarbonyl-3-(4-trifluoromethylphenyl)petanedioic acid 5-ethyl ester 1-ethyl ester (4n). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 4.03-3.91 (m, 5H), 3.75 (d, *J* = 10.0 Hz, 1H), 2.88 (dd, *J* = 16.0 Hz, *J* = 4.4 Hz, 1H), 2.75 (dd, *J* = 16.0 Hz, *J* = 10.0 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.09 (t, *J* = 7.2 Hz, 3H), 1.00 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.7, 167.7, 167.2, 144.1 (144.12), 144.1 (144.11), 129.7, 129.4, 128.7, 125.4, 125.3 (125.32), 125.3 (125.29), 12.3 (125.25), 122.7, 61.9, 61.5, 60.6, 56.9, 41.4, 38.3, 14.0, 13.9, 13.7; The enantiomeric excess was determined by HPLC with an AD-H column (*n*–hexane/*i*–PrOH = 90：10, flow rate 1 mL/min, λ = 210.5 nm), t_R = 9.77 min (major), t_R = 16.30 min (minor), 94% ee.

References

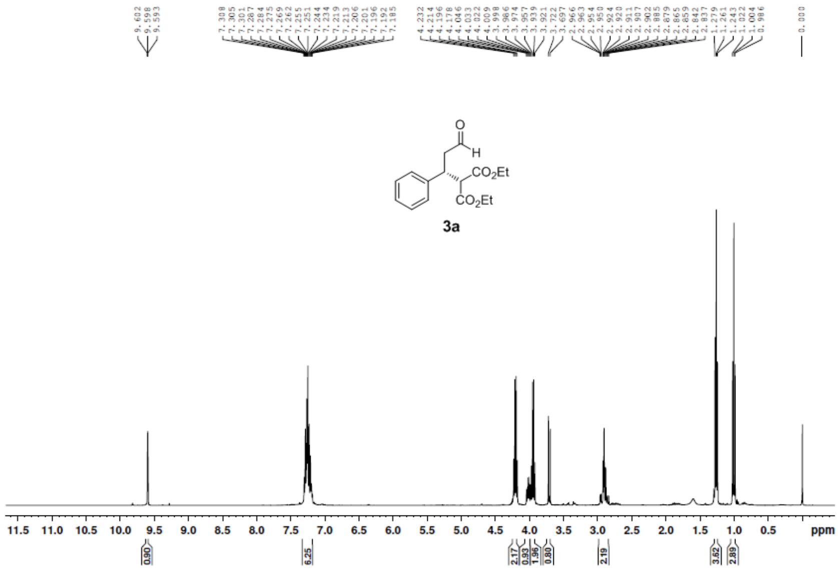
1). E. A. Krasnokutskaya, N. I. Semenischeva, V. D. Filimonov and P. Knochel, *Synthesis.*, 2007, **1**, 81-84.

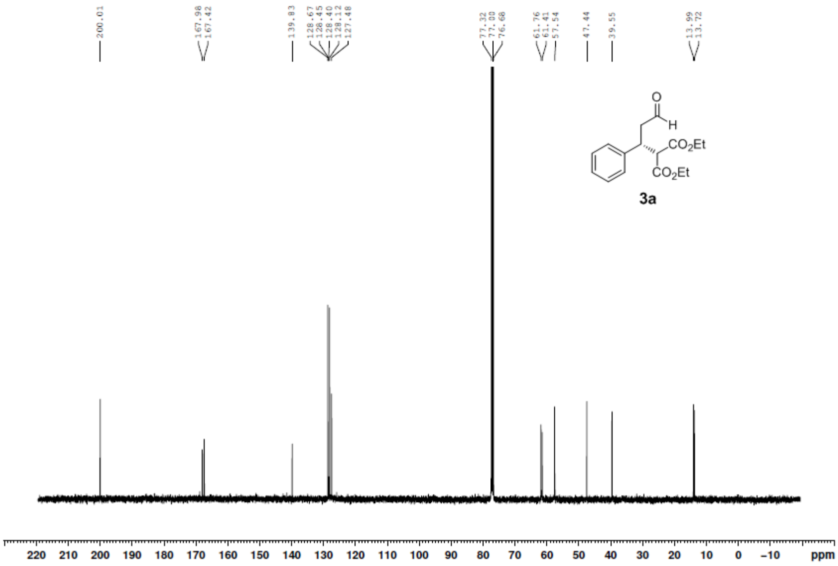
2). K. E. Torraca, S. I. Kuwabe and S. L. Buchwald, *J. Am. Chem. Soc.*, 2000, **122**, 12907-12908.

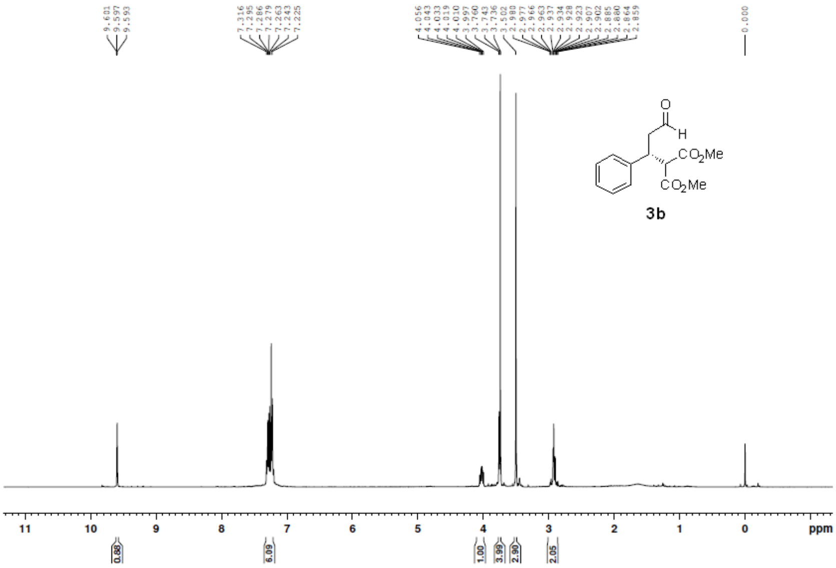
3). The absolute configuration of the products **3** were confirmed by comparing the $[\alpha]_D$ values with those of the reported known compounds **3a-c**.[4]

4). S. Brandau, A. Landa, J. Franzén, M. Marigo and K. A. Jøgensen, *Angew. Chem. Int. Ed.*, 2006, **45**, 4305 -4309.

NMR spectrogram



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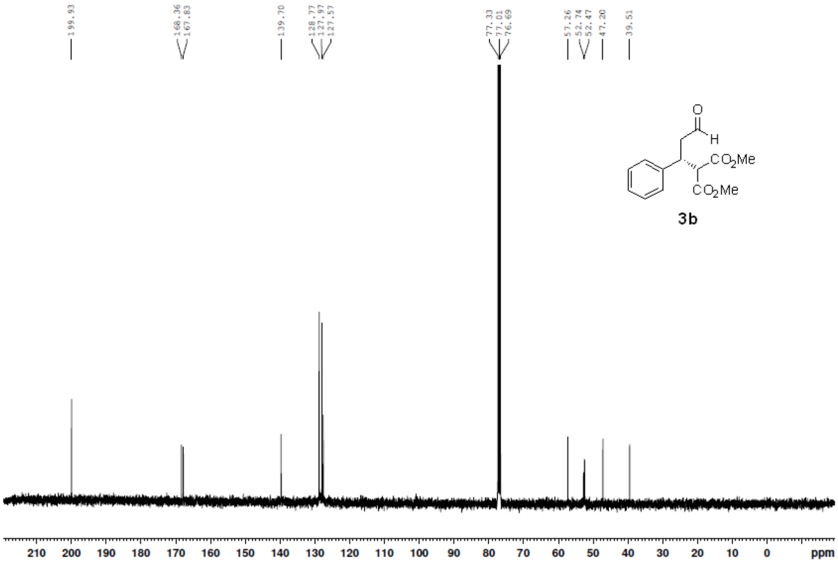
2.90
2.05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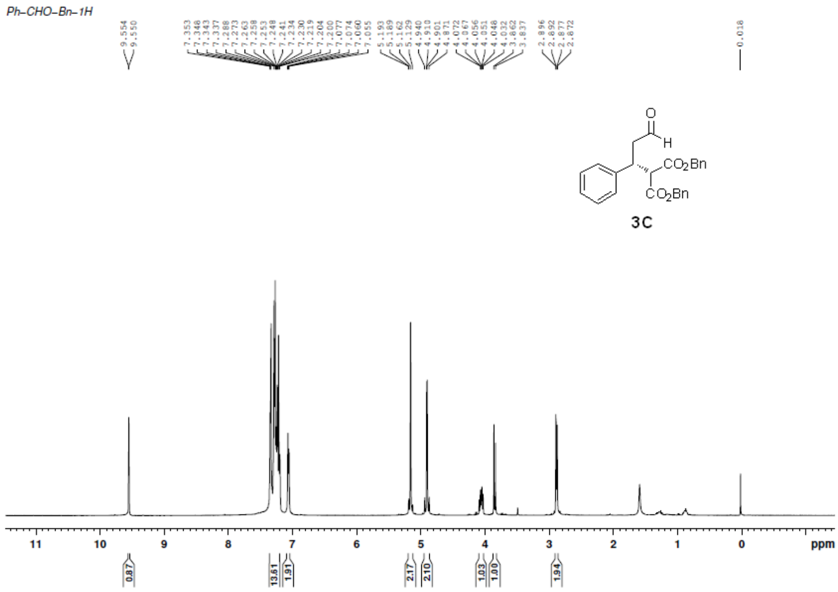
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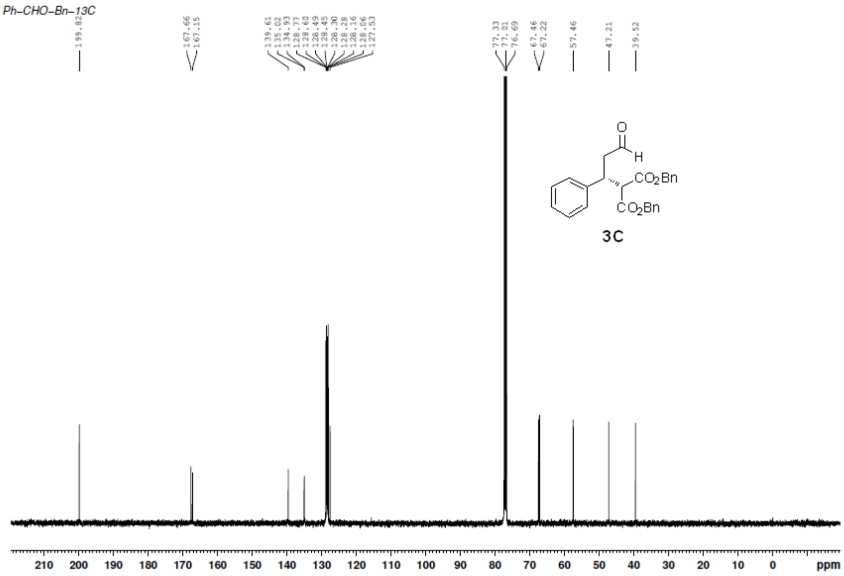
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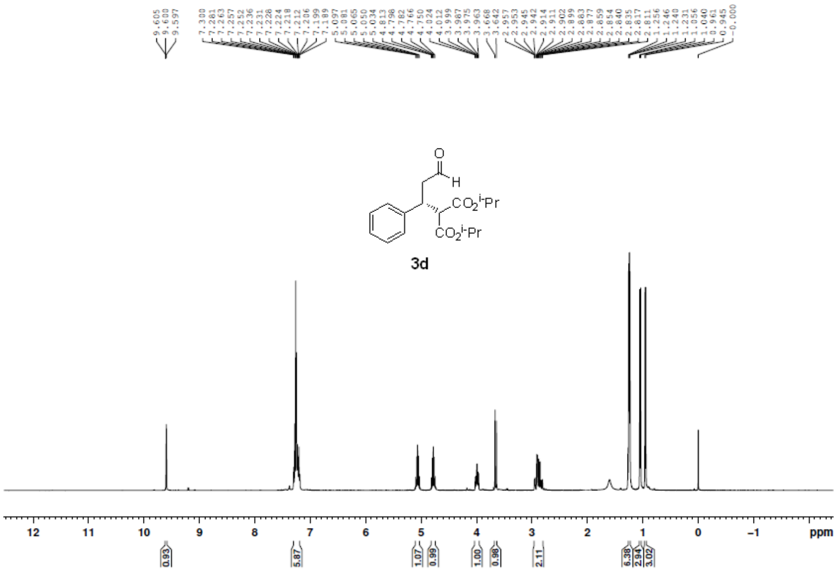
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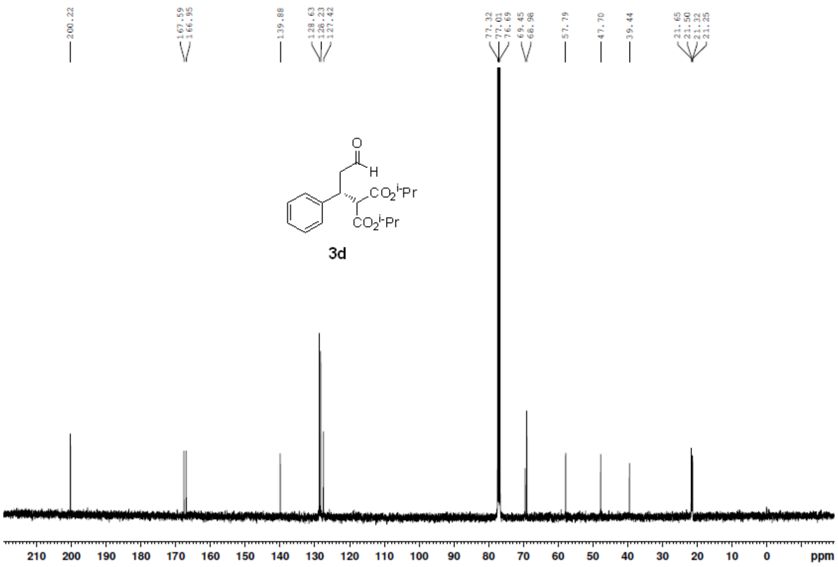
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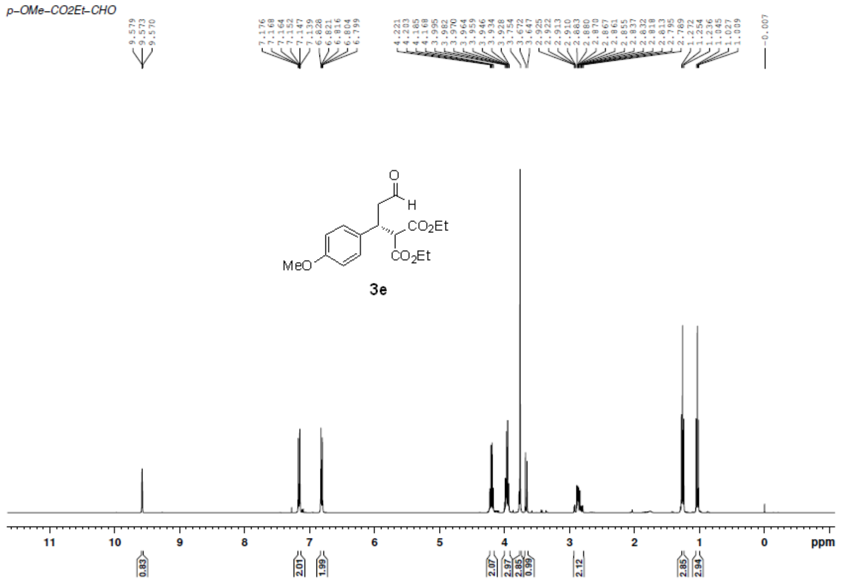

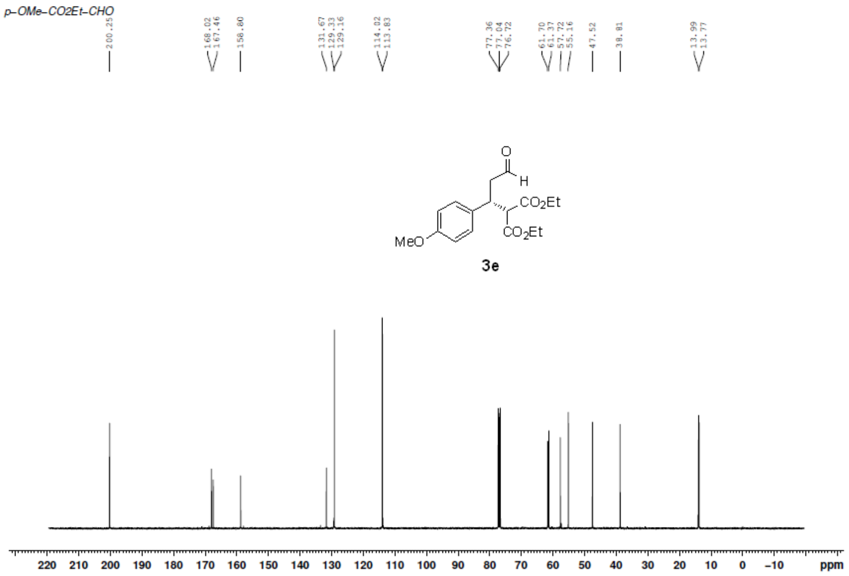

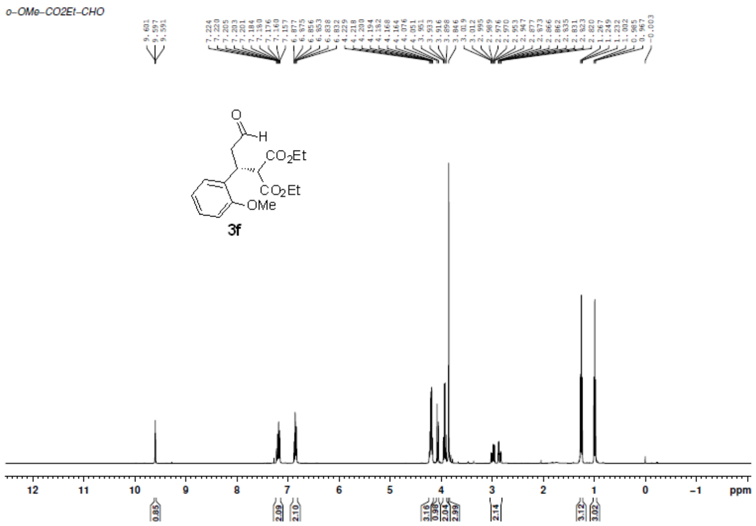
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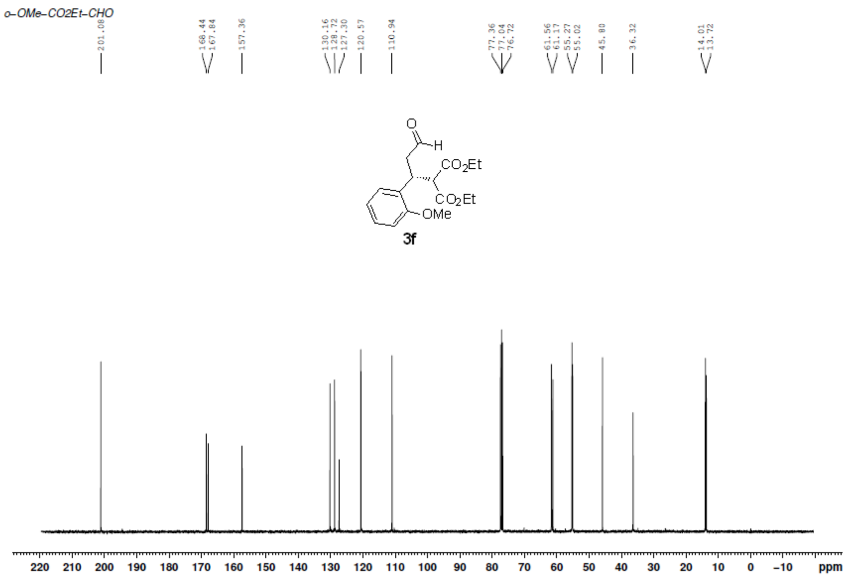
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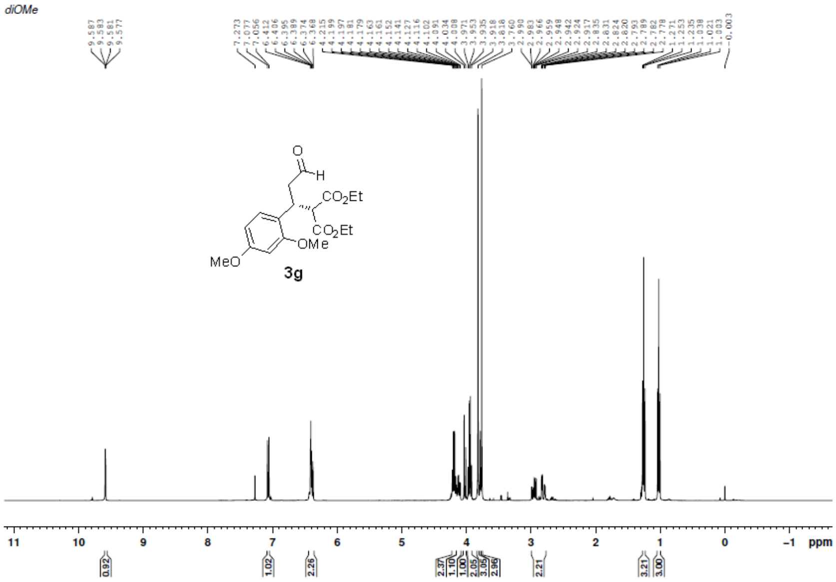
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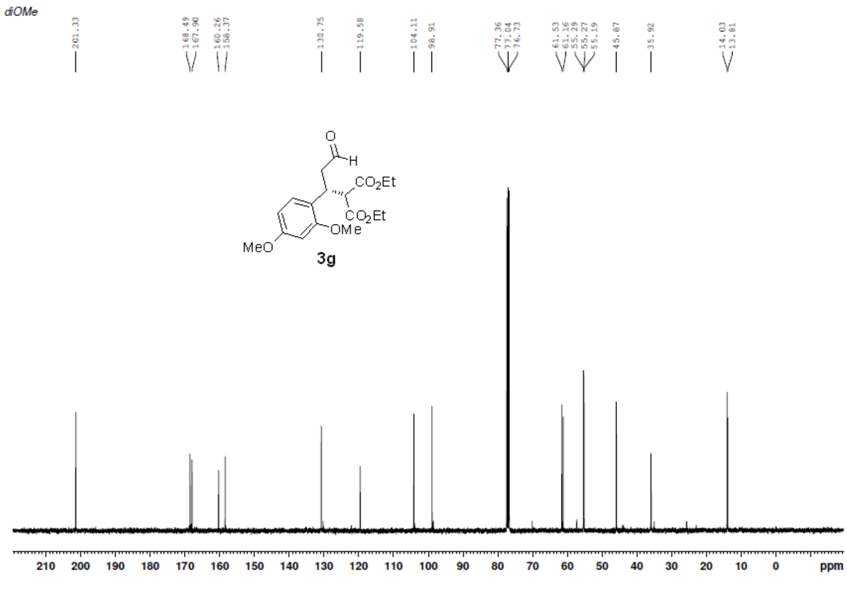
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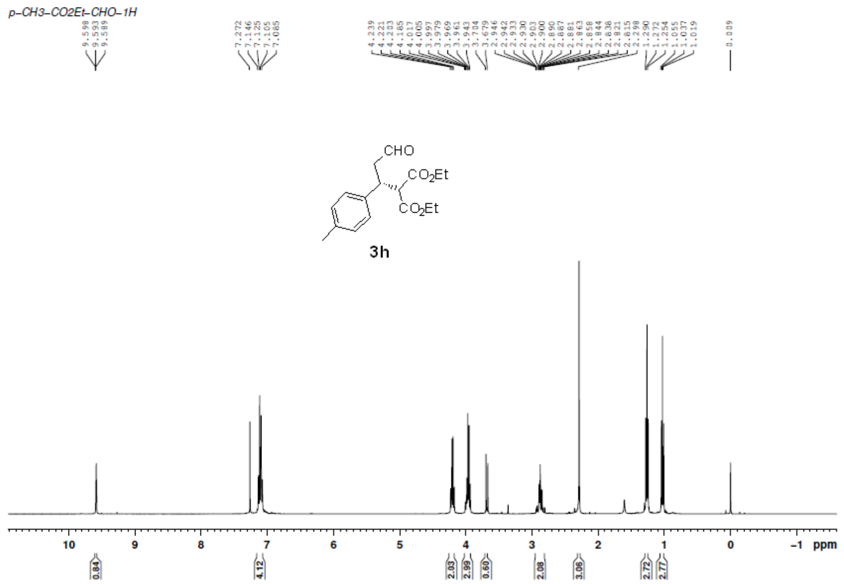
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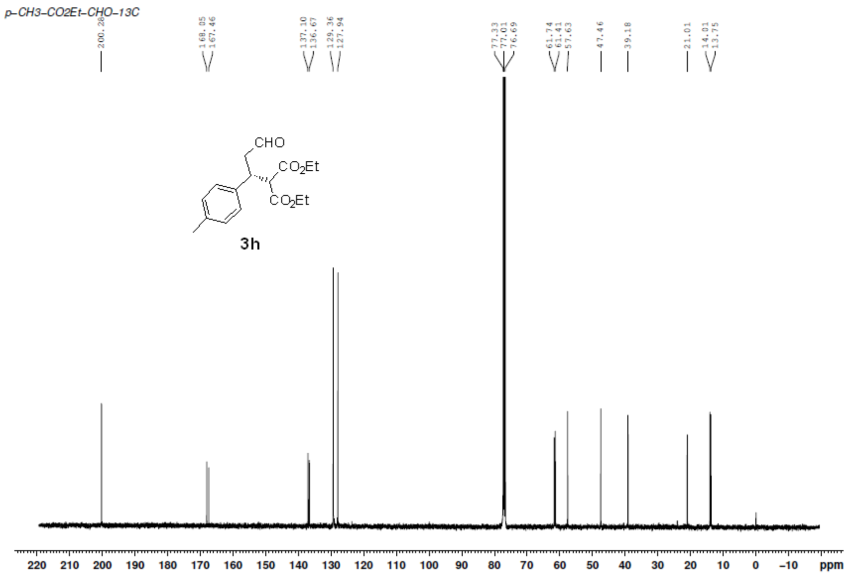
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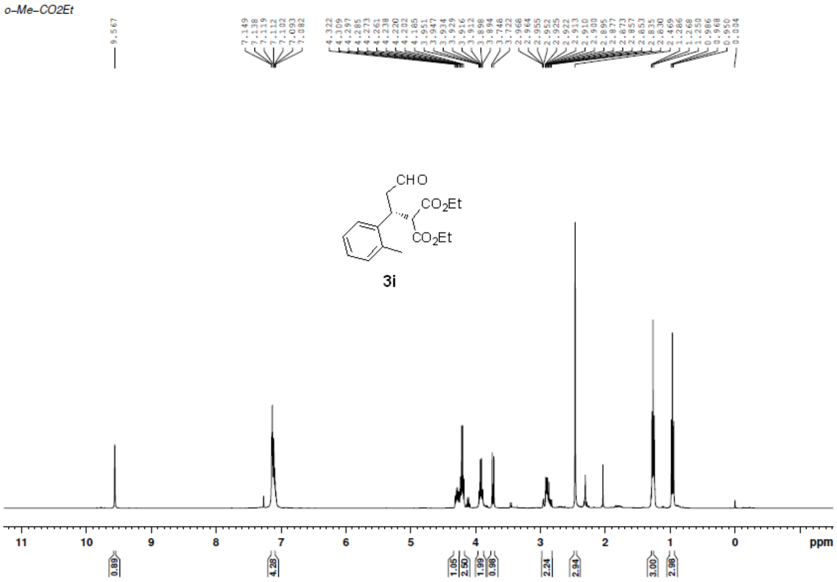
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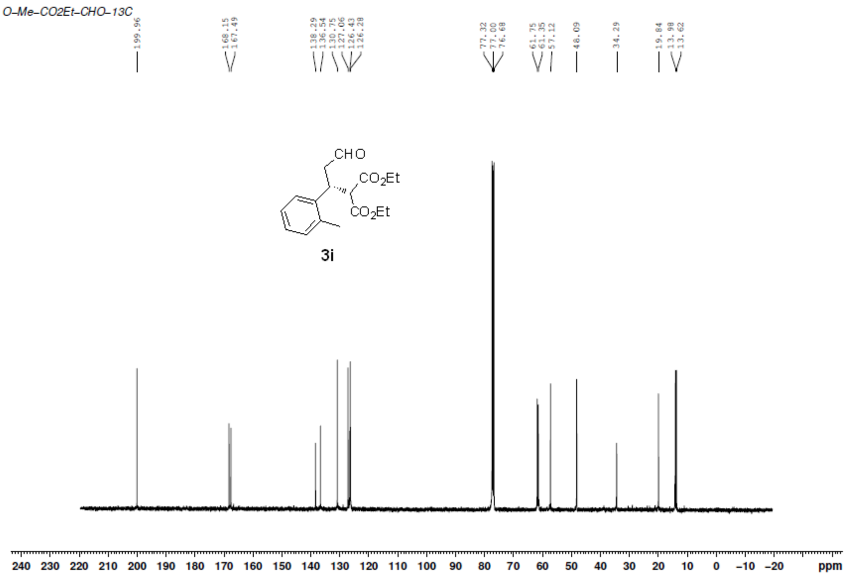
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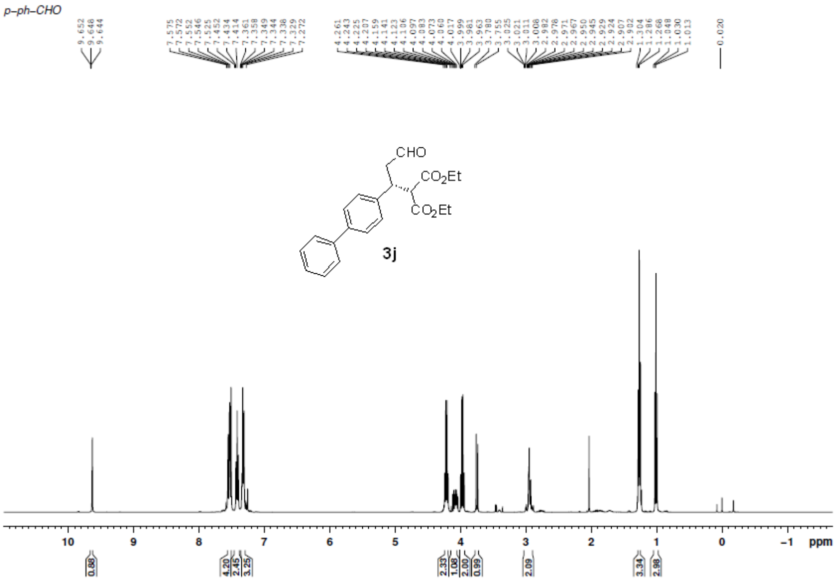
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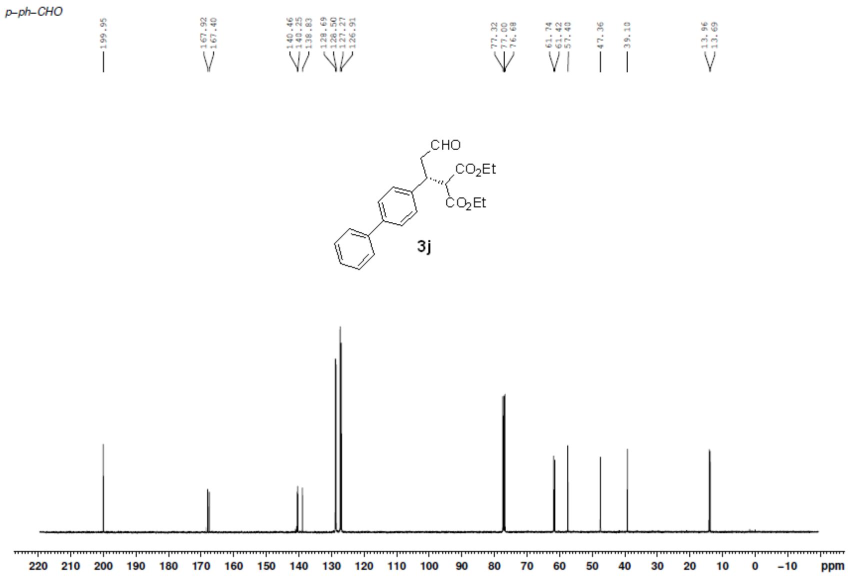
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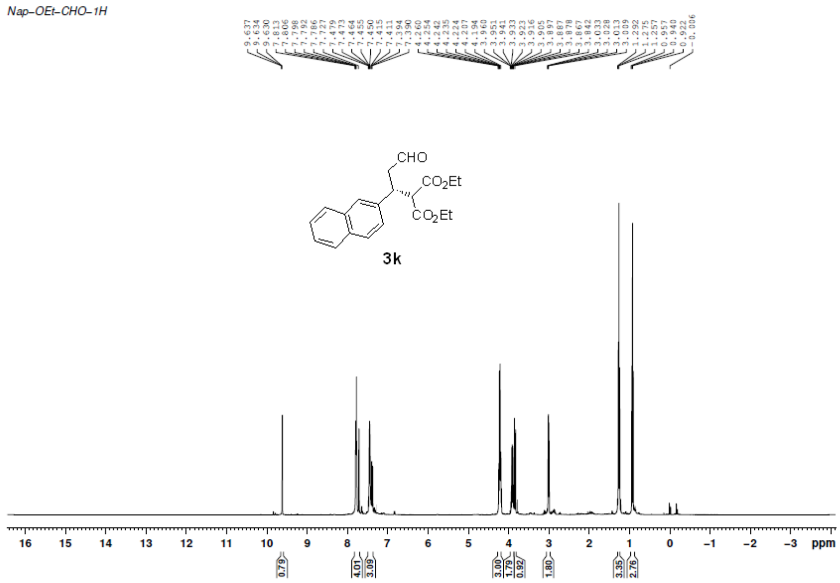
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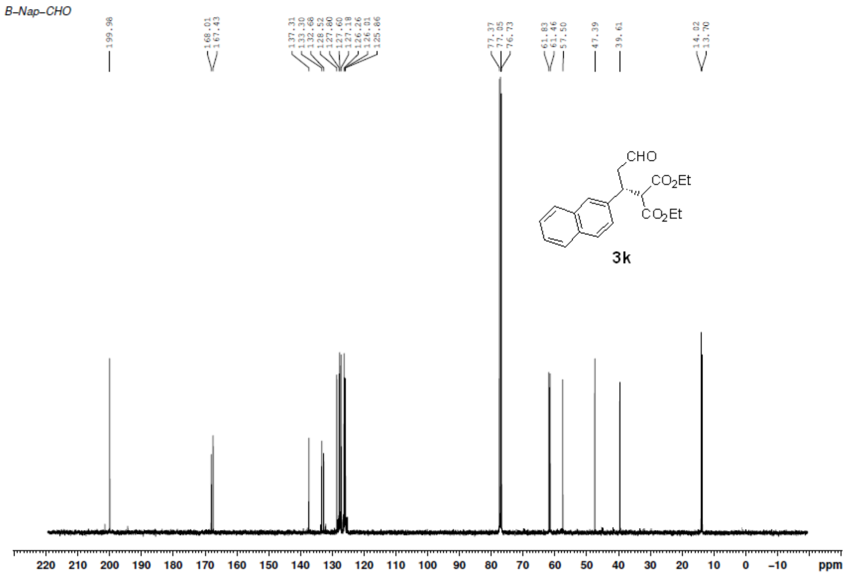
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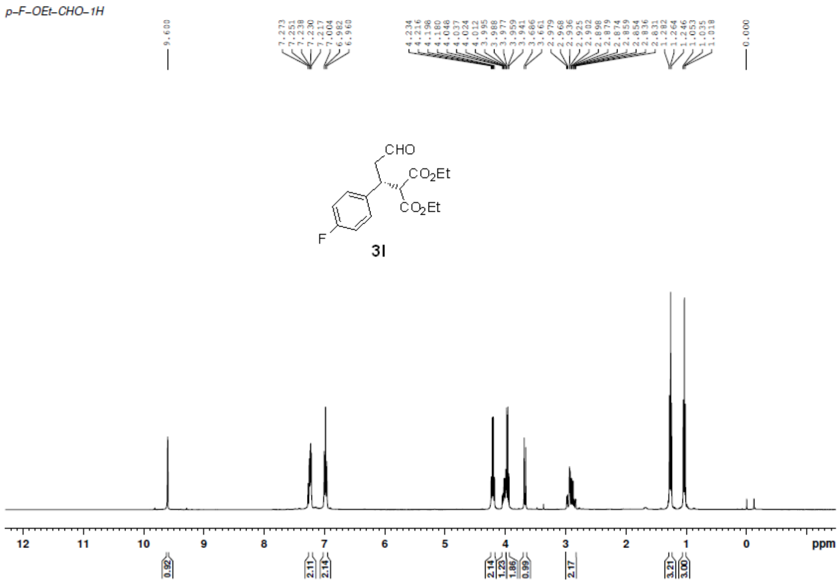
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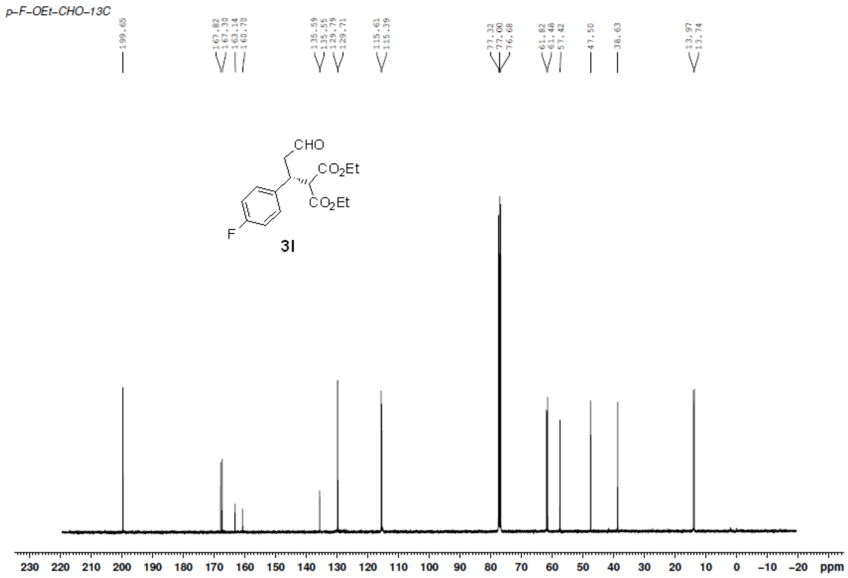
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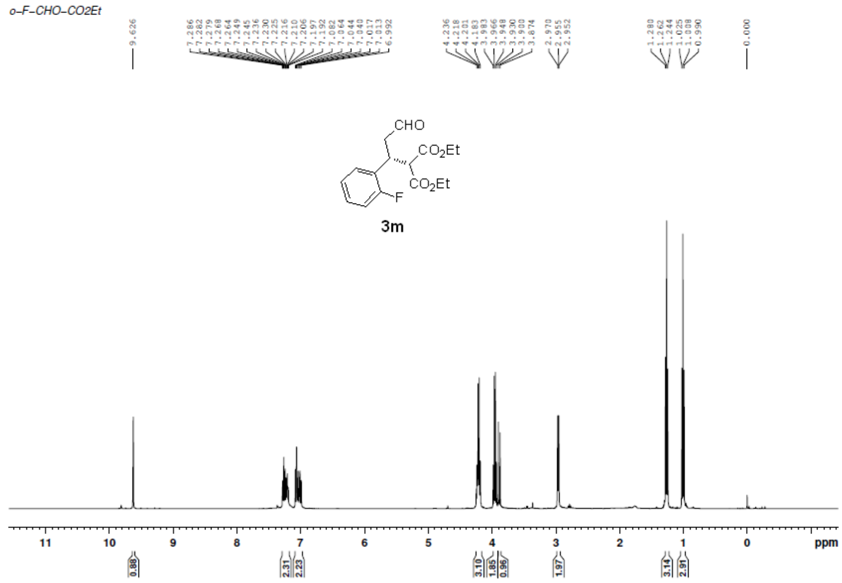
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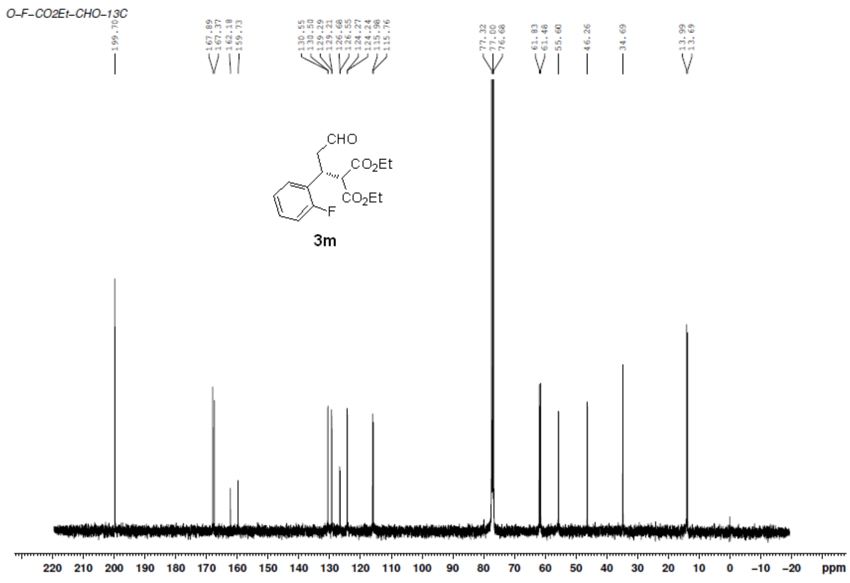
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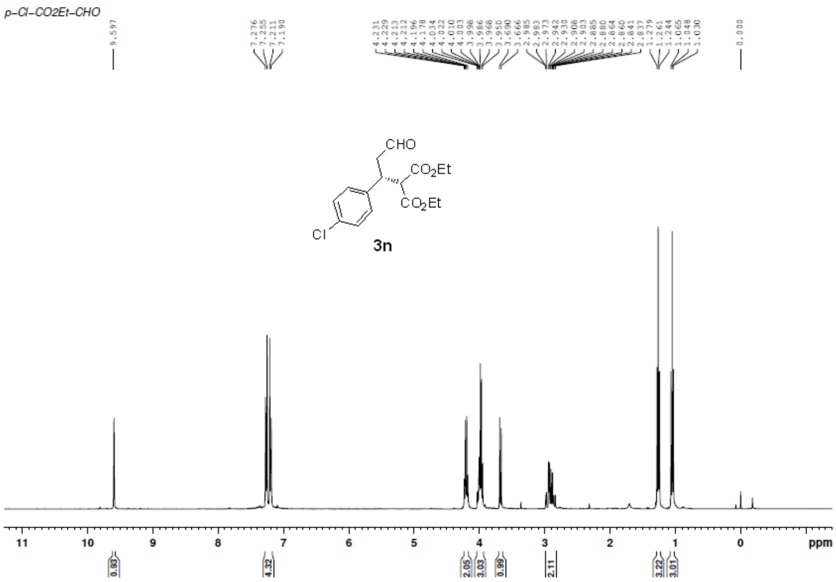

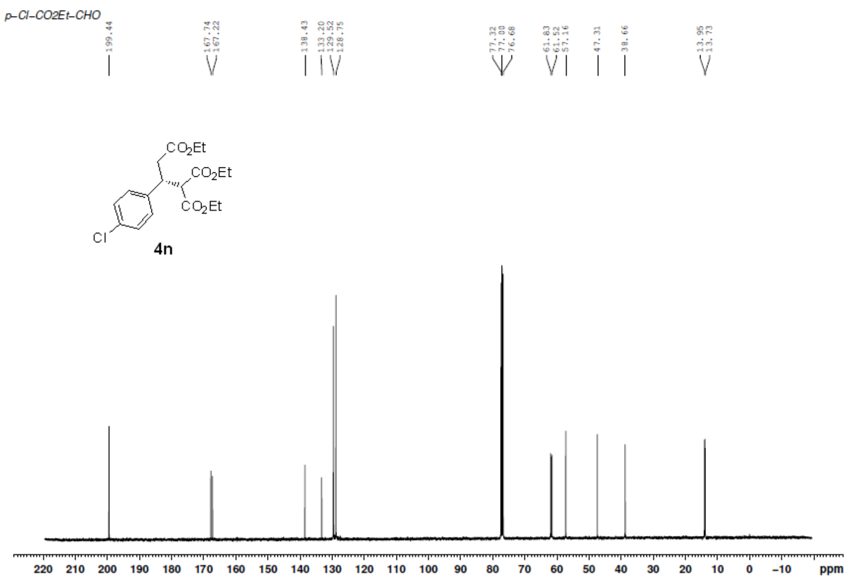

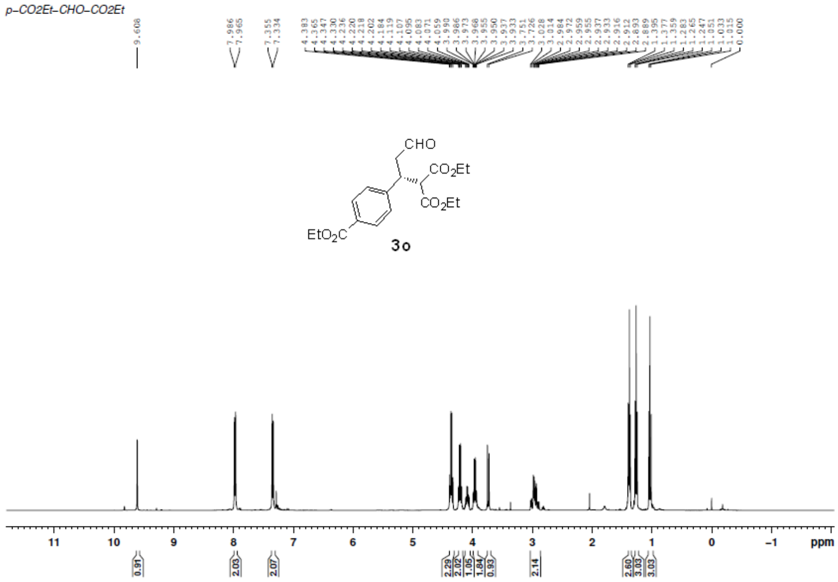
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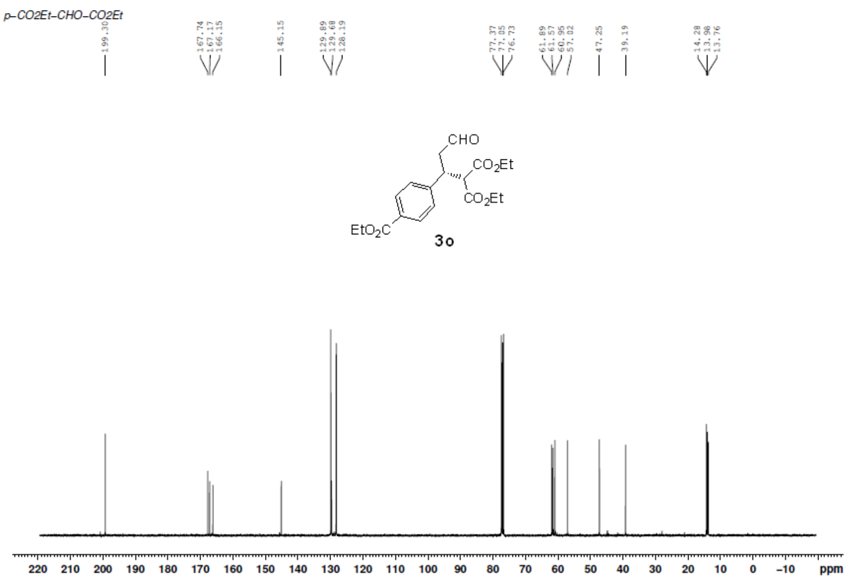
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129.89
129.68
128.19
77.37
77.05
76.73
61.89
61.57
60.95
57.02
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39.19
14.28
13.98
13.76
CHO
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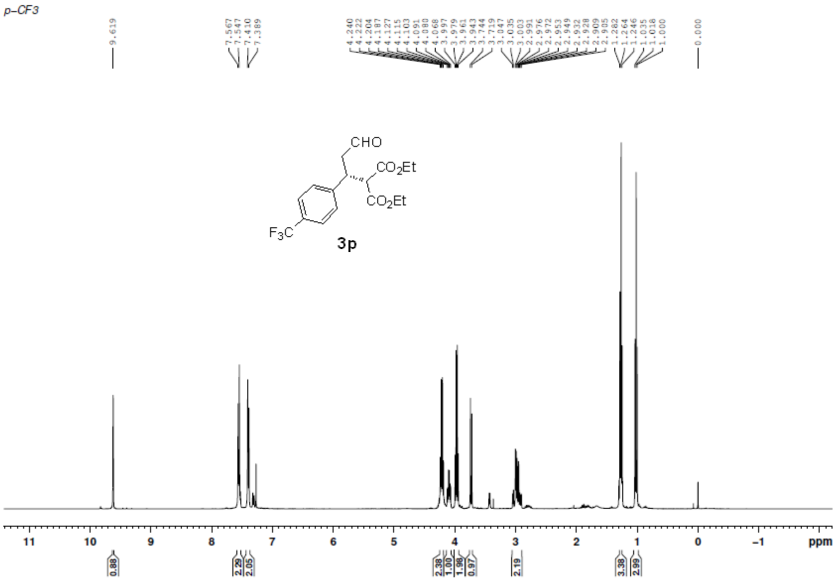
CO2Et
EtO2C
3o
ppm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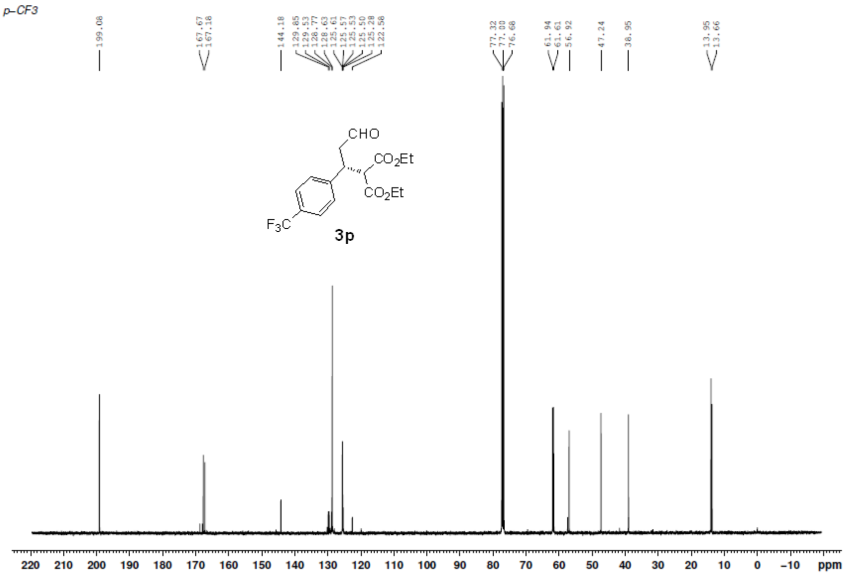
CHO
CO2Et
CO2Et
F3C
3p
ppm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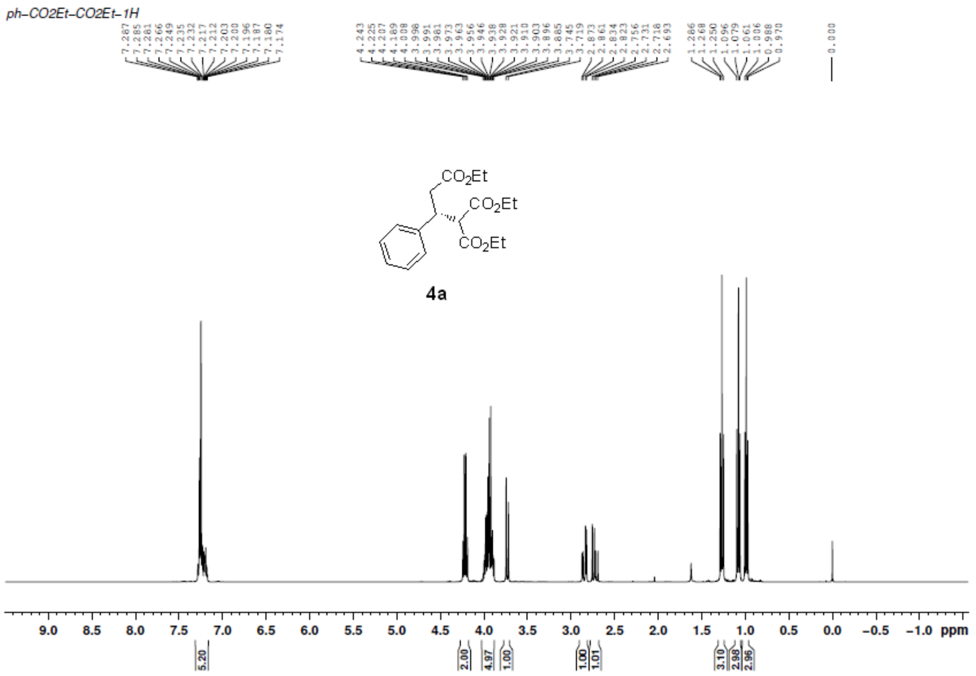
CHO
CO2Et
CO2Et
F3C
3p
ppm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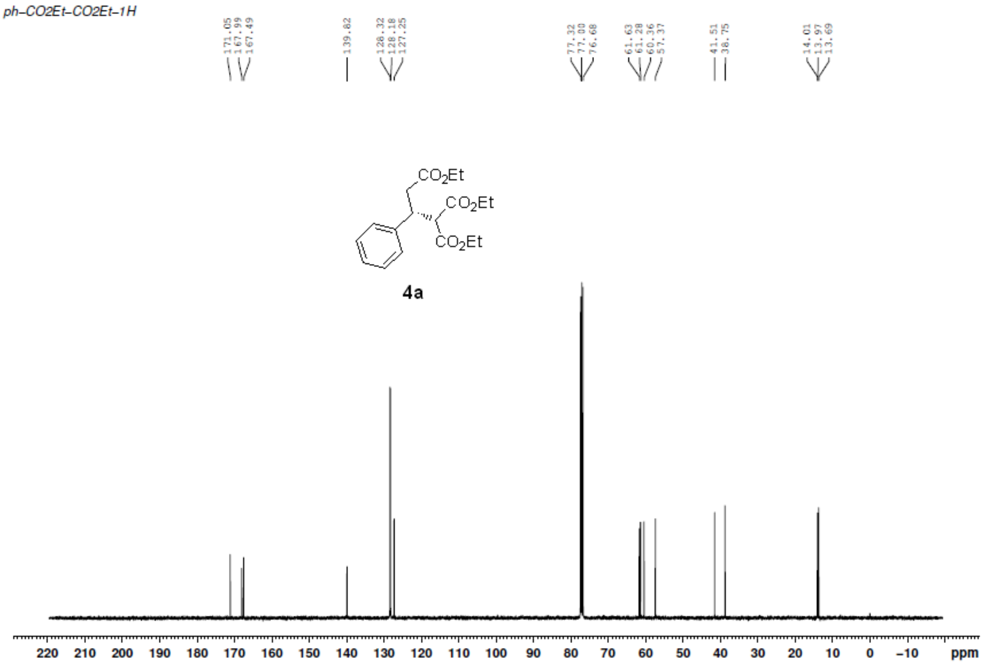
CO2Et
CO2Et
4a
ppm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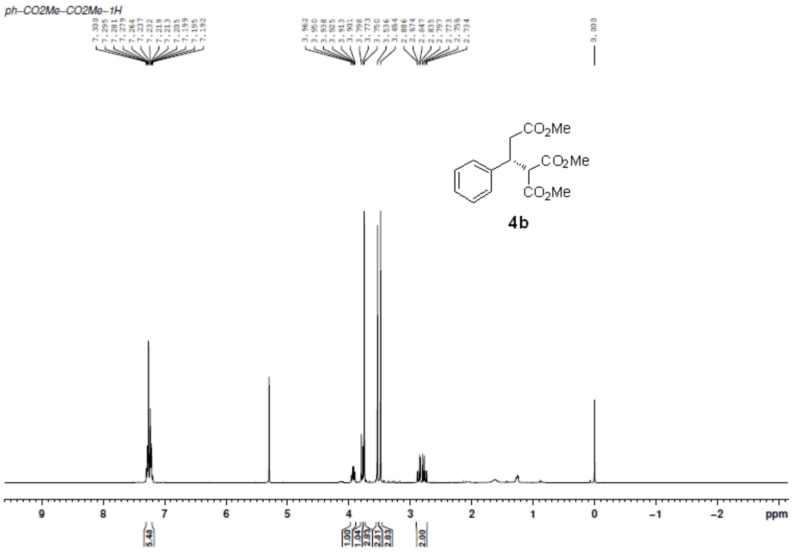
CO2Et
CO2Et
4a
ppm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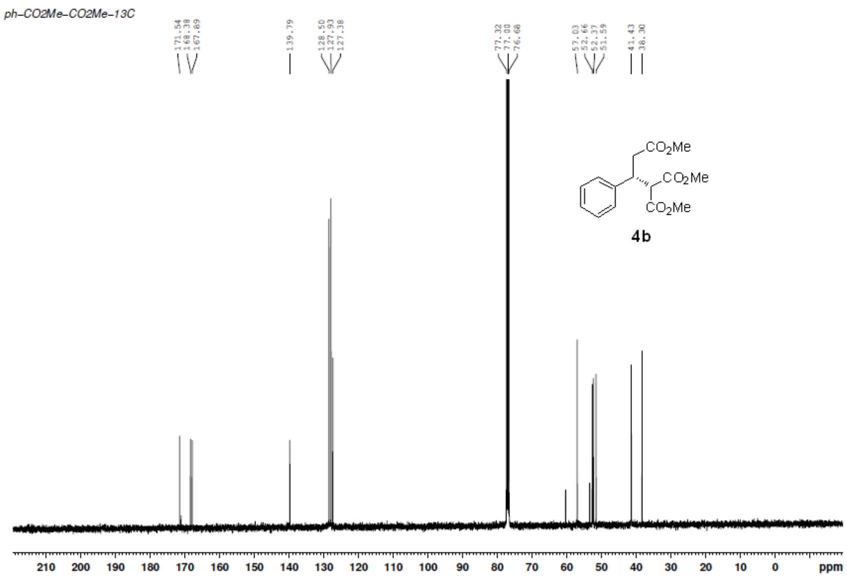
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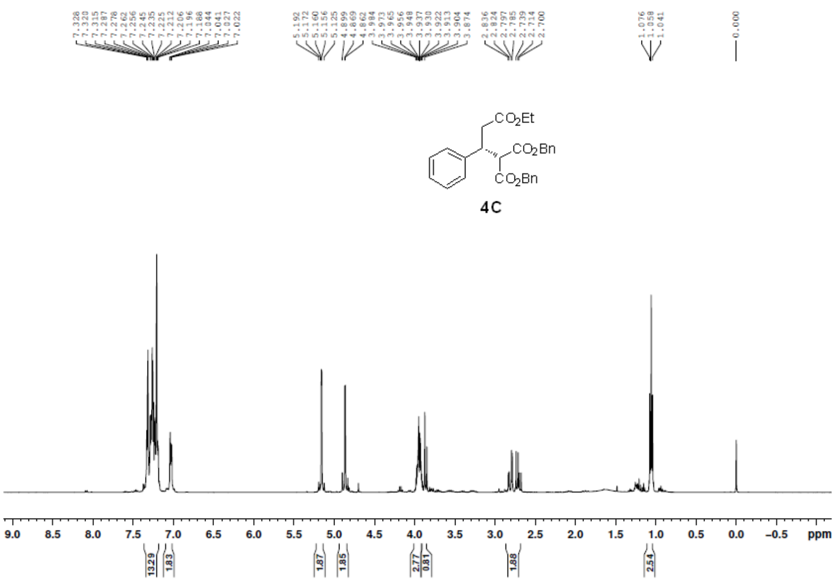
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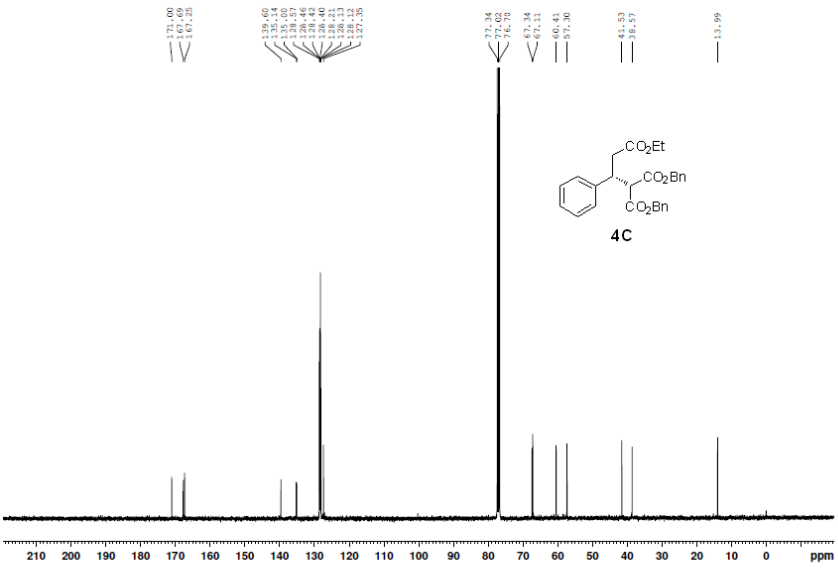
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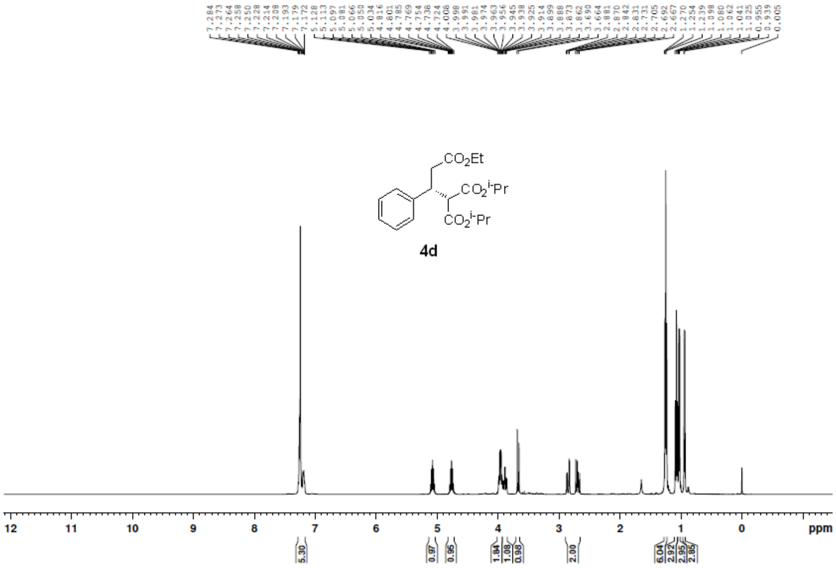
CO2Bn
CO2Bn
4C
ppm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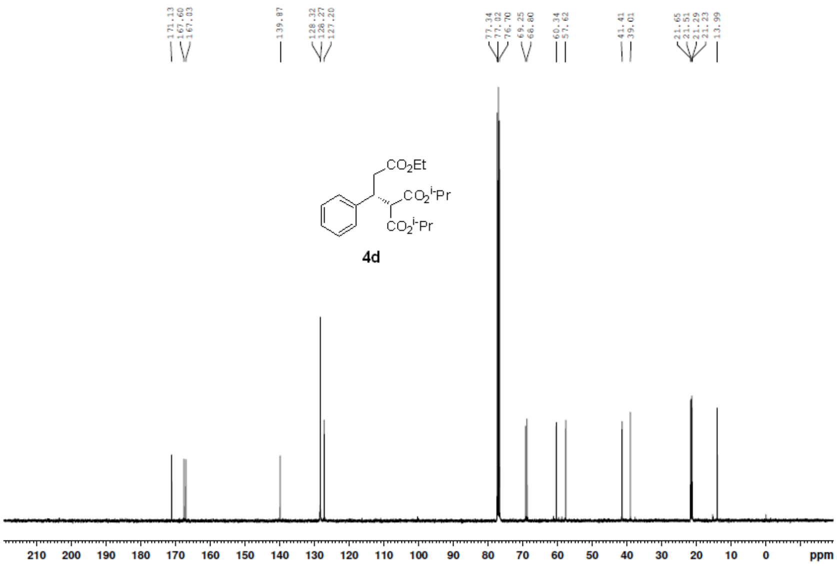
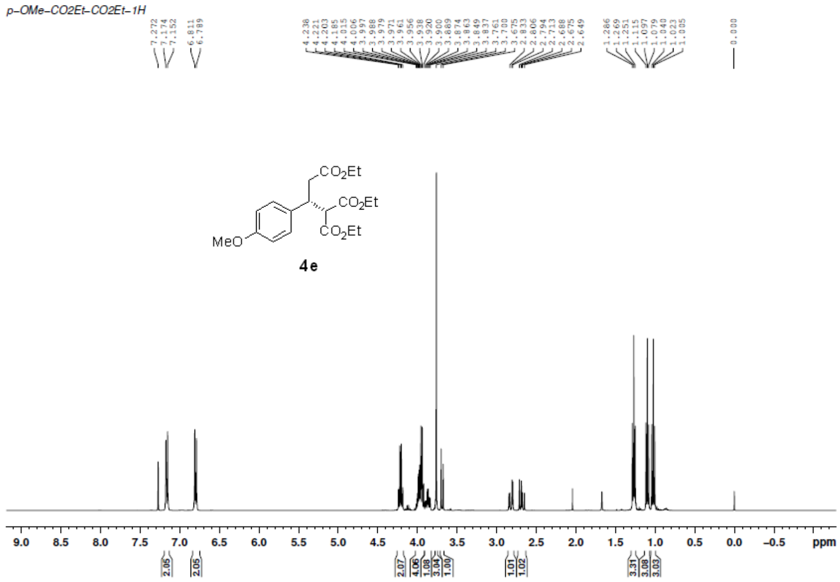
CO2iPr
CO2iPr
4d
ppm


CO2Et
CO2iPr
CO2iPr
4d
ppm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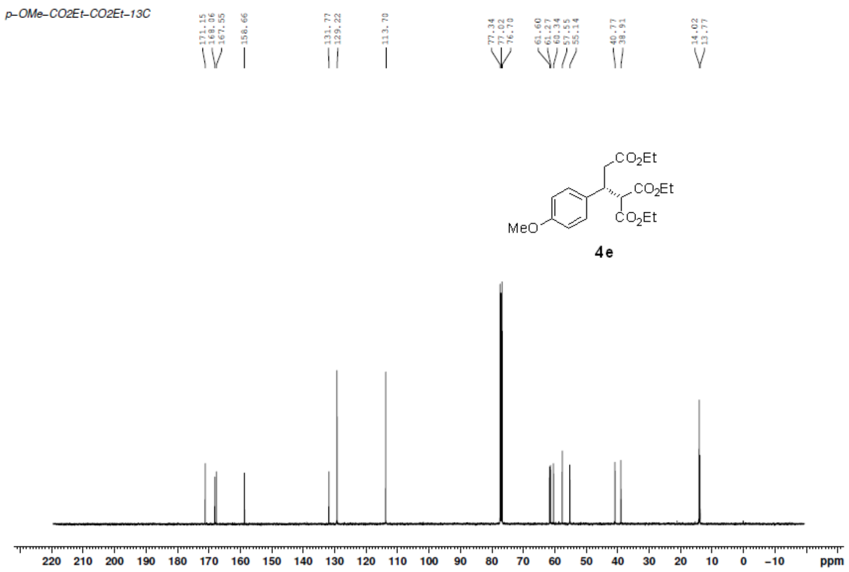
4e
ppm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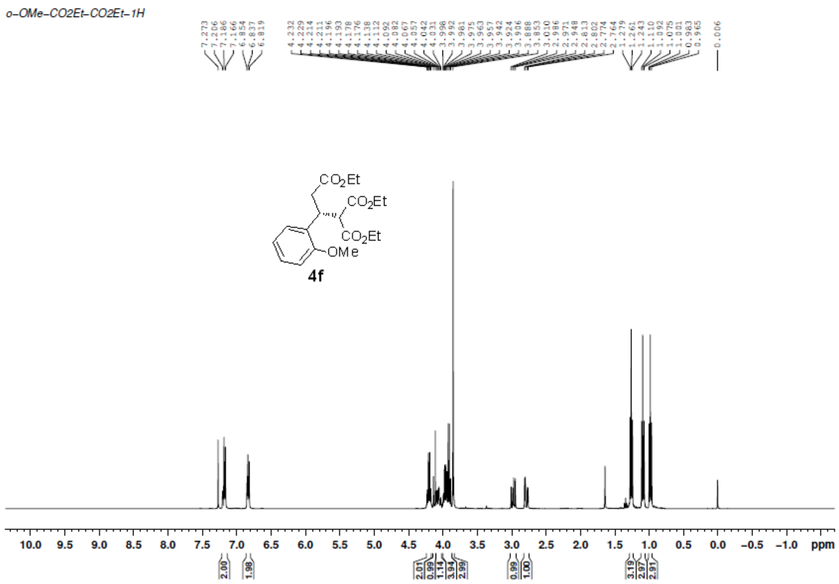
4e
ppm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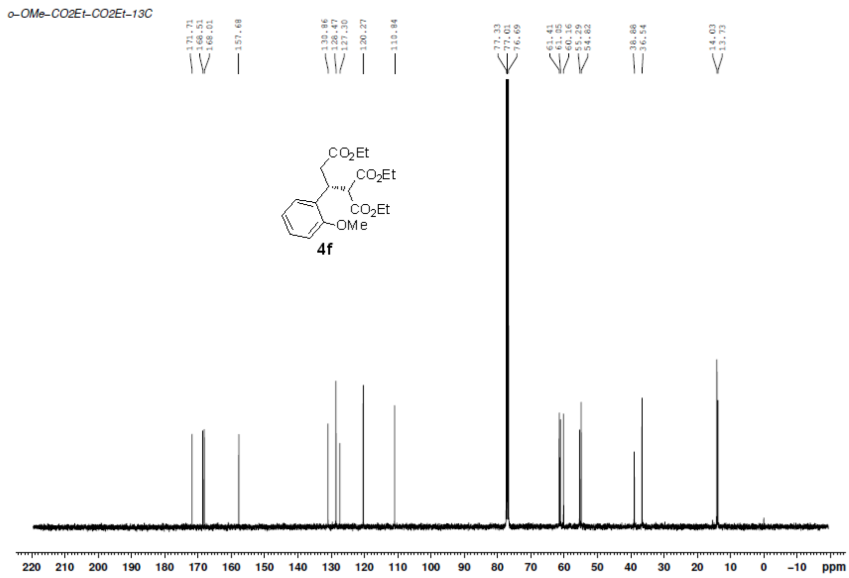
CO2Et
CO2Et
CO2Et
OMe
4f
ppm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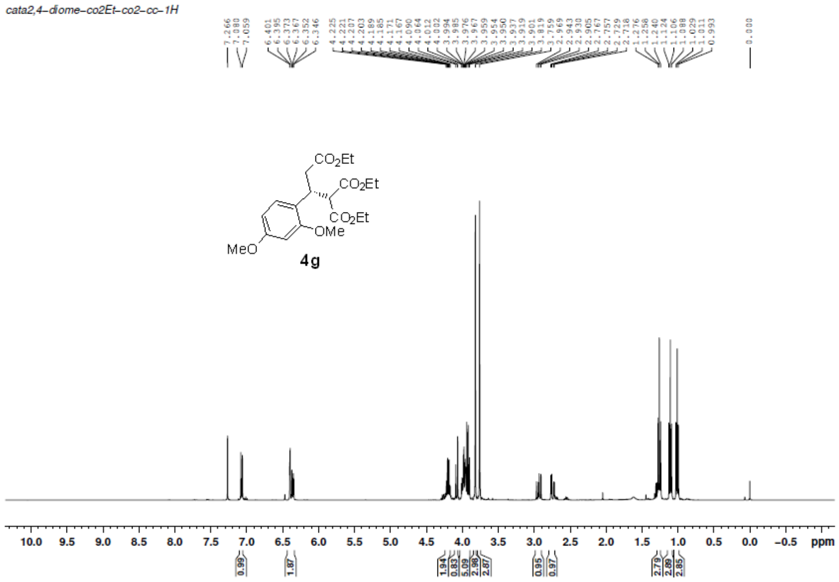
CO2Et
CO2Et
CO2Et
OMe
4f
ppm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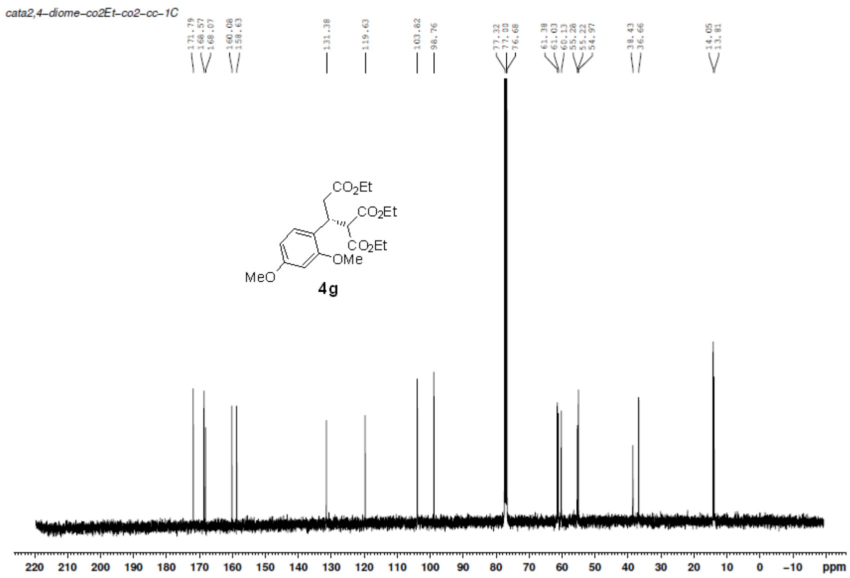
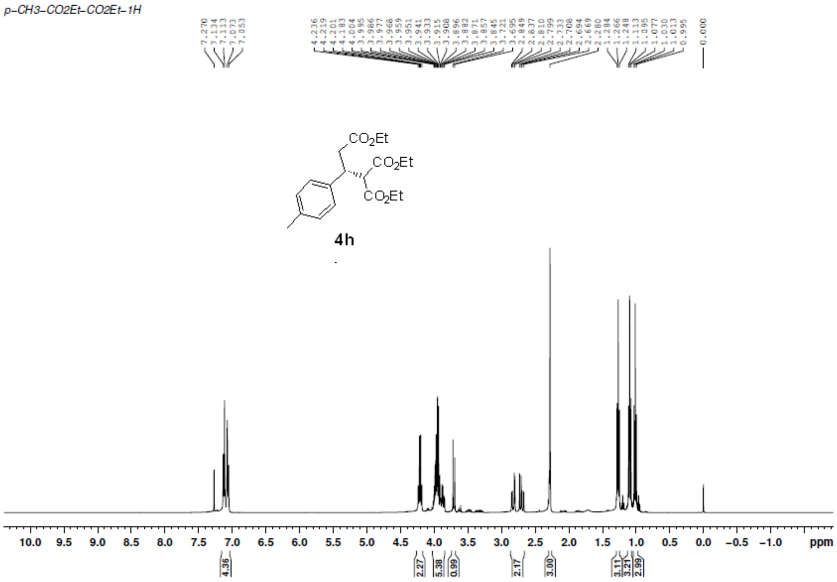
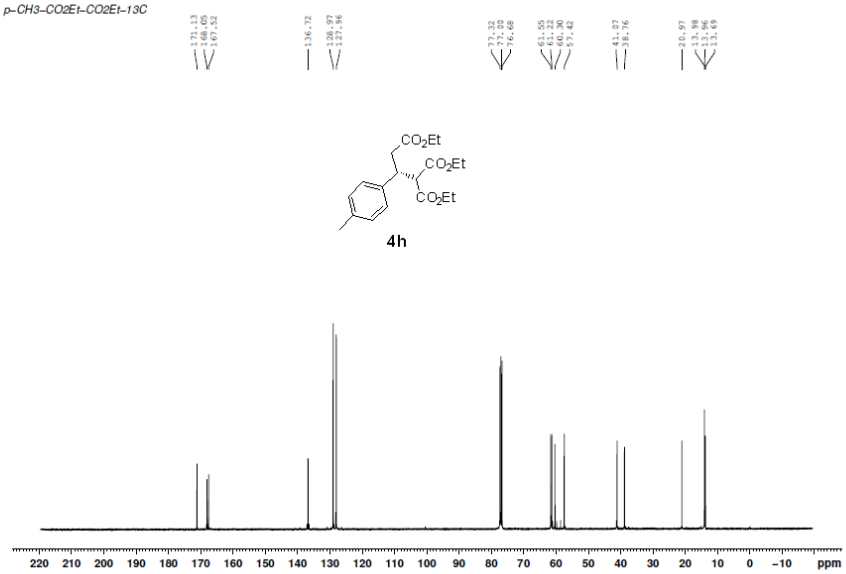
CO2Et
CO2Et
CO2Et
OMe
MeO
4g
ppm

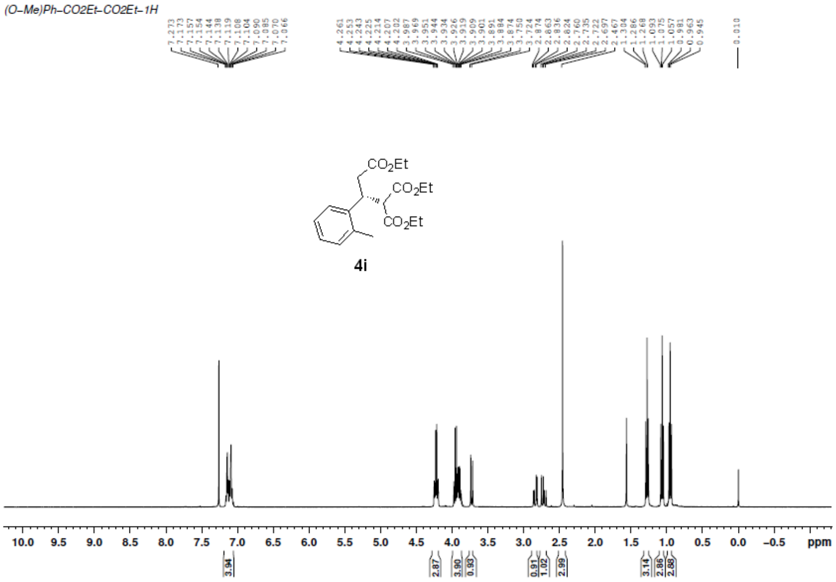

cata2,4-diome-co2Et-co2-cc-1C
CO2Et
CO2Et
CO2Et
OMe
MeO
4g
ppm

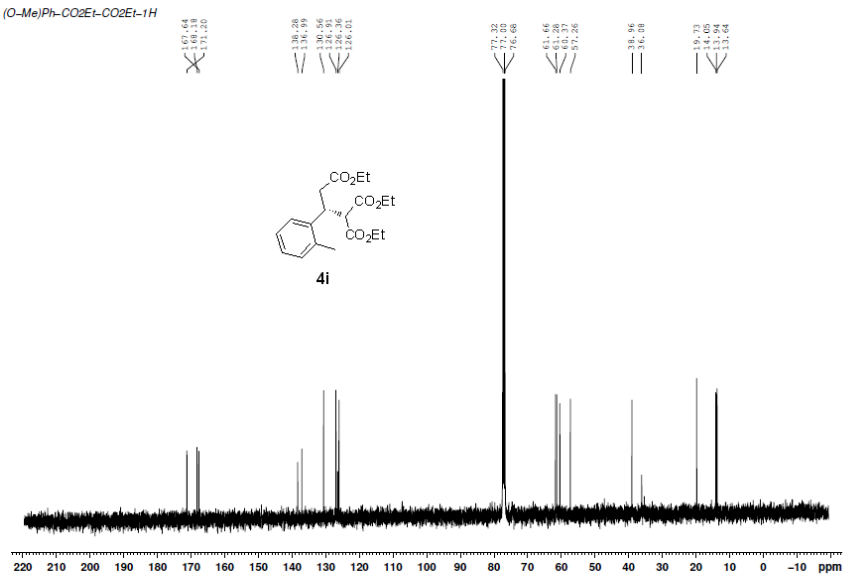
p-CH3-CO2Et-CO2Et-1H

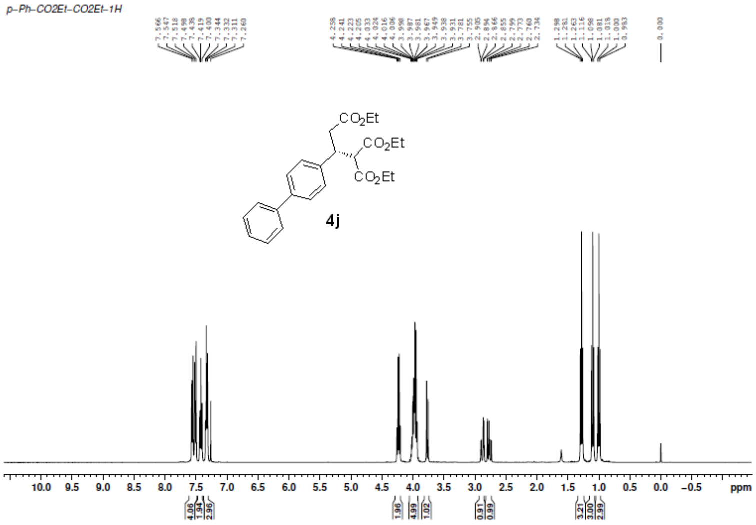


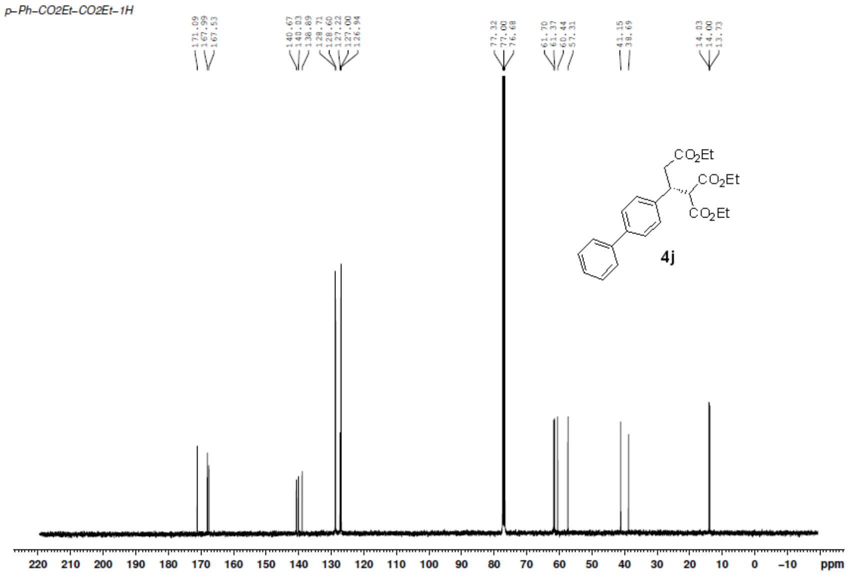
p-CH3-CO2Et-CO2Et-13C

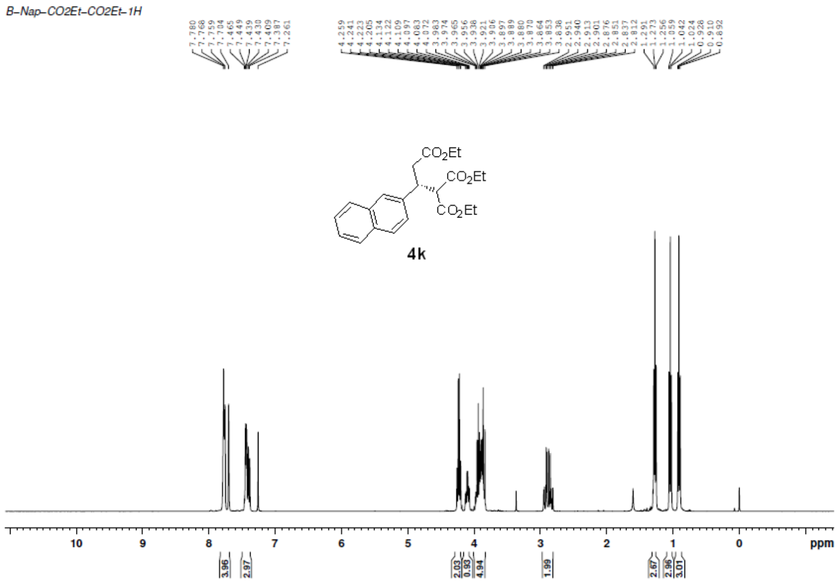


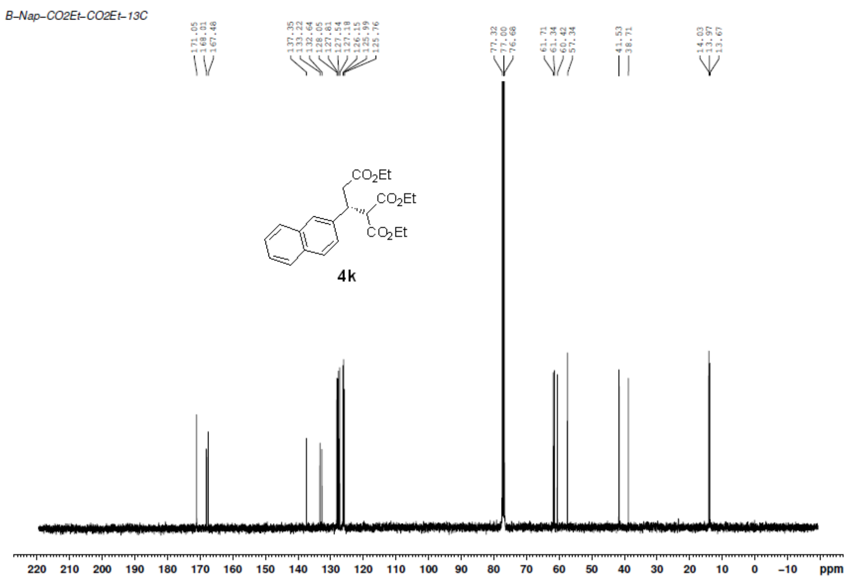

(O-Me)Ph-CO2Et-CO2Et-1H
CO2Et
CO2Et
CO2Et
4i
ppm

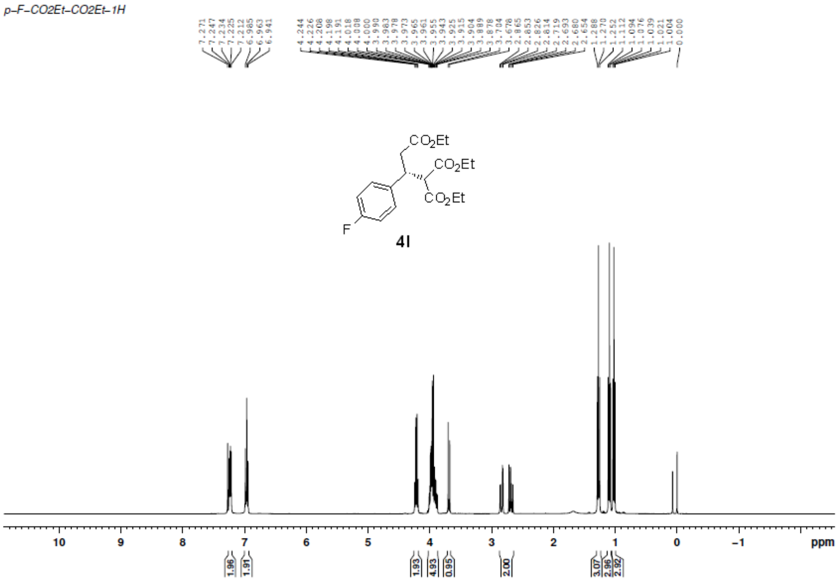

(O-Me)Ph-CO2Et-CO2Et-1H
CO2Et
CO2Et
CO2Et
4i
ppm






B-Nap-CO2Et-CO2Et-1H
4k
ppm


B-Nap-CO2Et-CO2Et-13C
4k
ppm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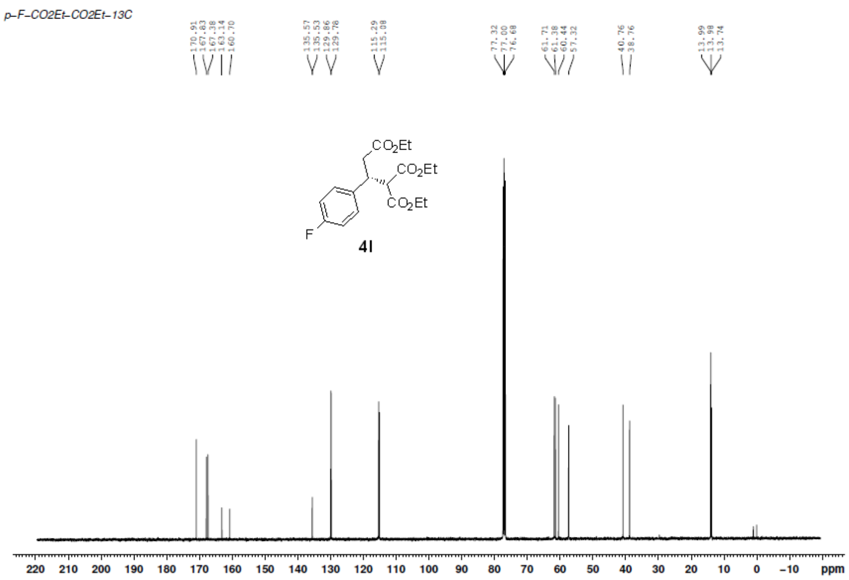
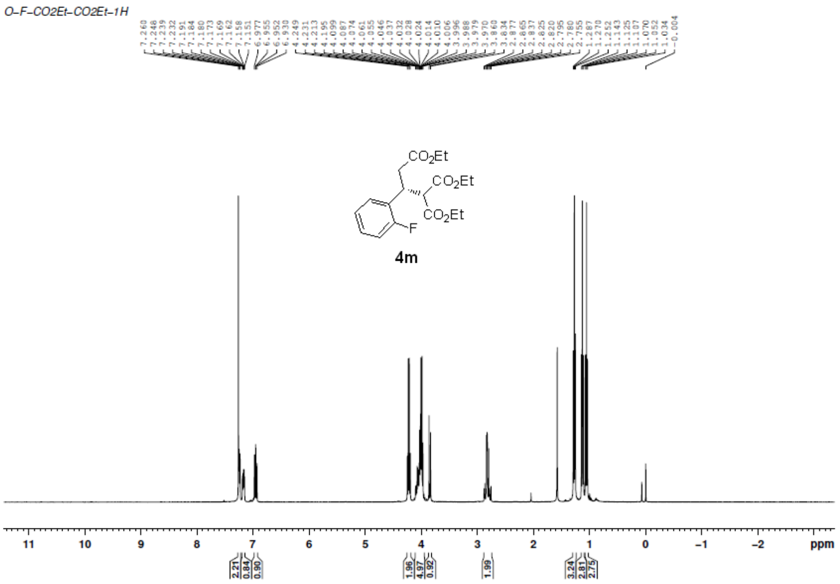
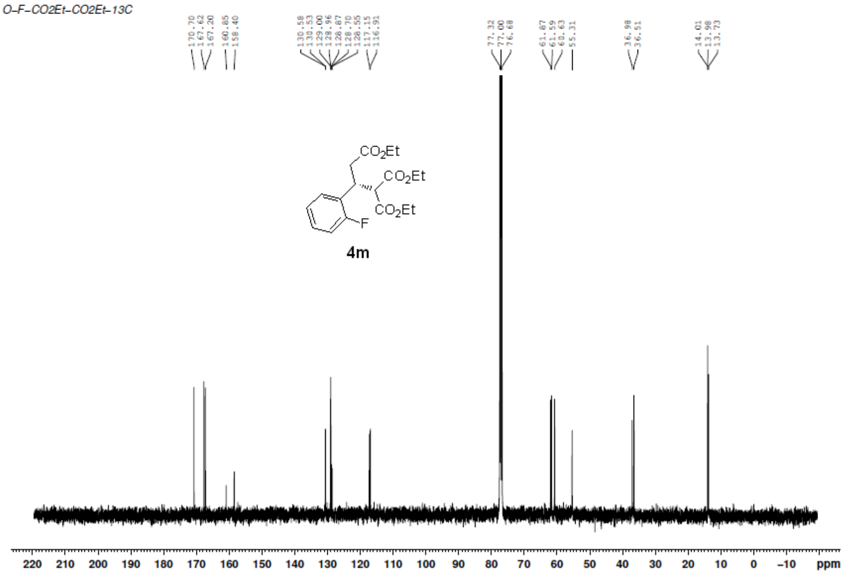
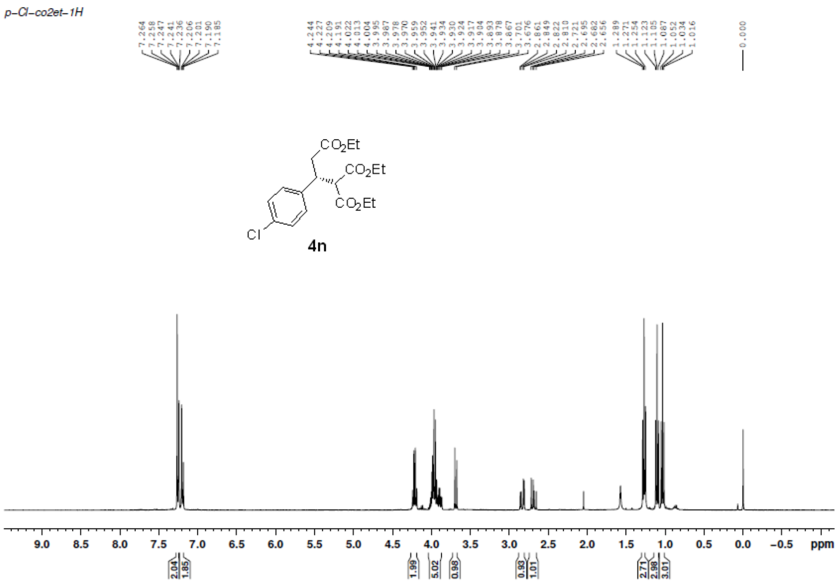
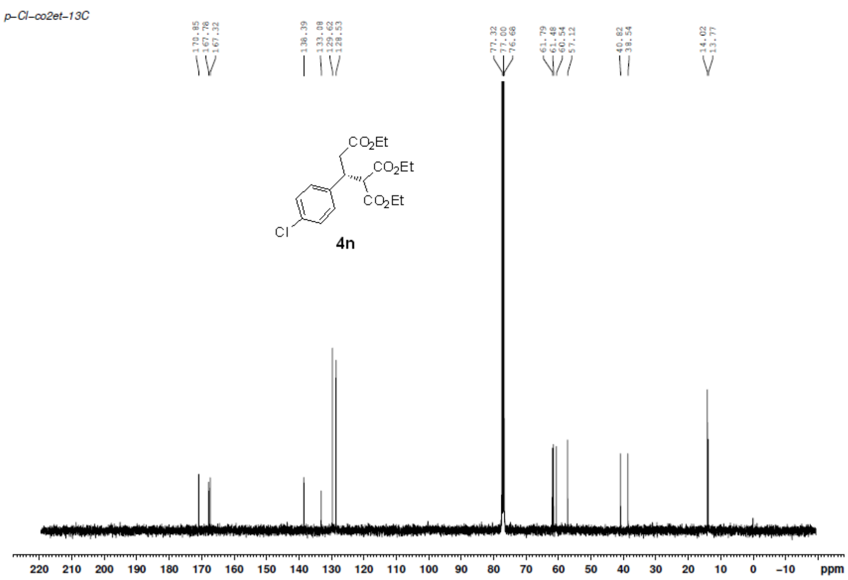
CO2Et
CO2Et
CO2Et
F
4l
ppm

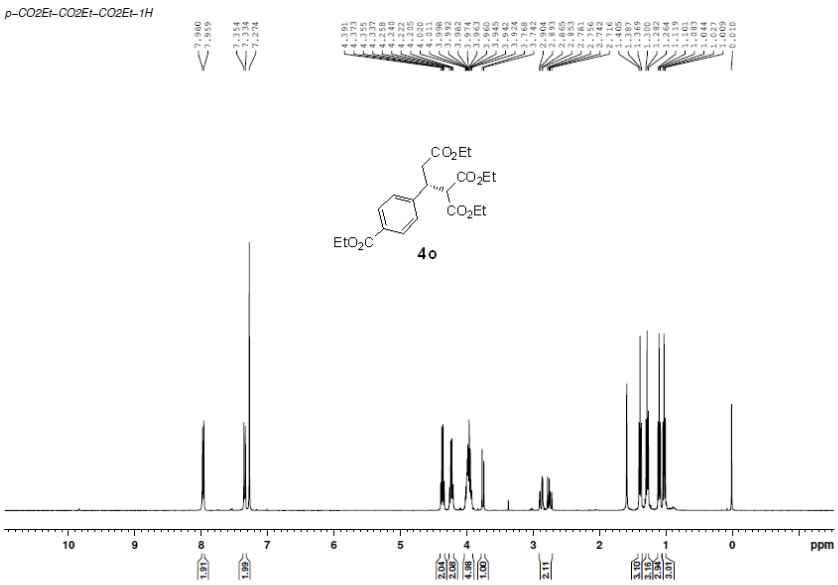

p-F-CO2Et-CO2Et-13C
CO2Et
CO2Et
CO2Et
F
4l
ppm

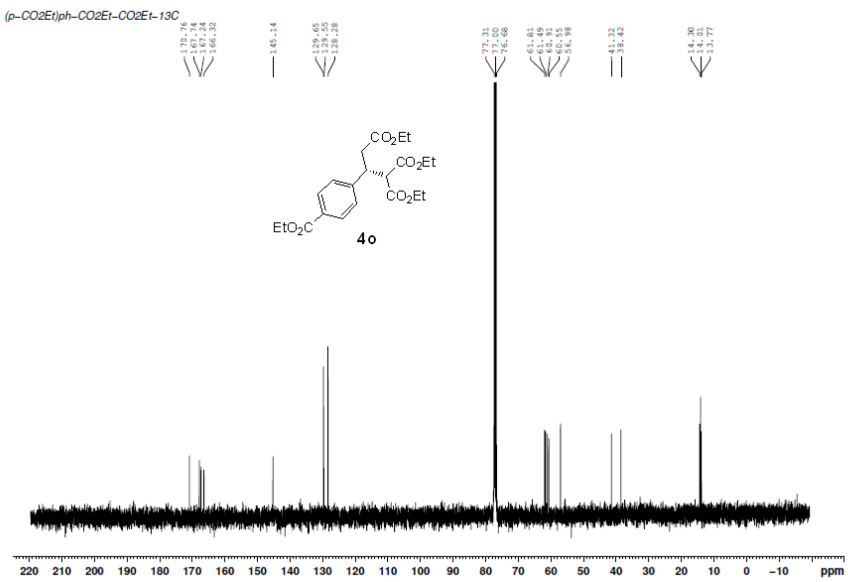


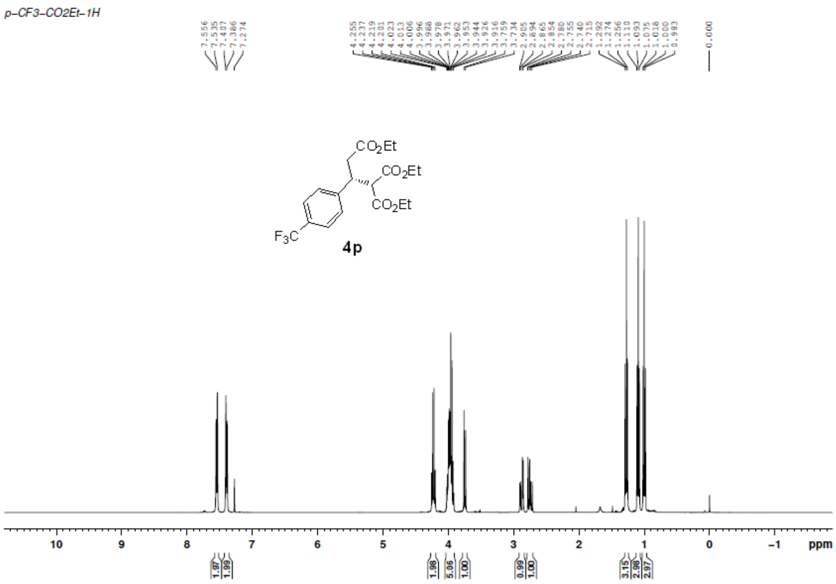


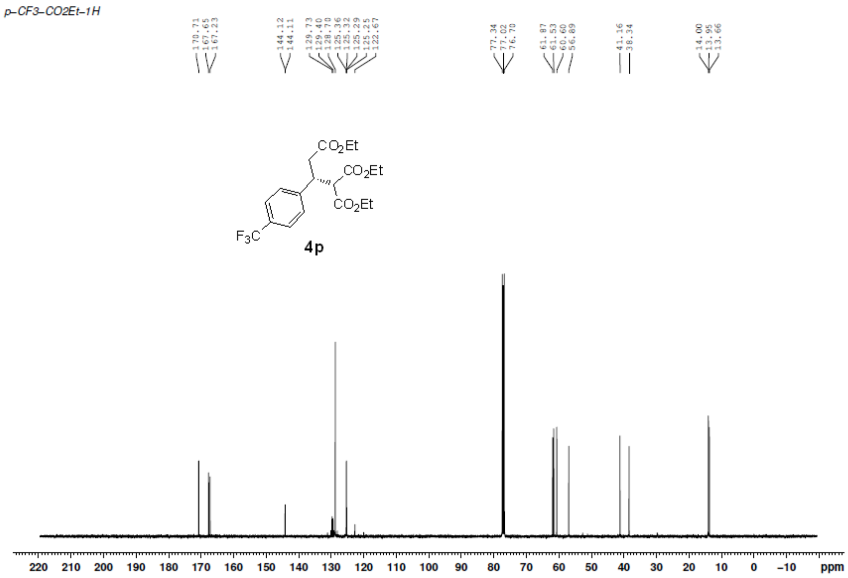

p-Cl-co2et-1H
CO2Et
CO2Et
CO2Et
Cl
4n
ppm


p-Cl-co2et-13C
CO2Et
CO2Et
CO2Et
Cl
4n
ppm


p–CO2Et–CO2Et–CO2Et–1H
CO2Et
CO2Et
CO2Et
EtO2C
4o
ppm

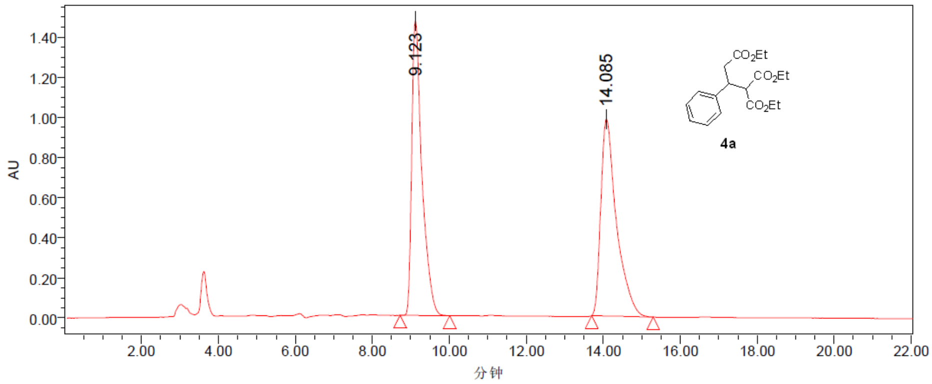

(p–CO2Et)ph–CO2Et–CO2Et–13C
CO2Et
CO2Et
CO2Et
EtO2C
4o
ppm


p–CF3–CO2Et–1H
CO2Et
CO2Et
CO2Et
F3C
4p
ppm


p–CF3–CO2Et–1H
CO2Et
CO2Et
CO2Et
F3C
4p
ppm

HPLC Spectra

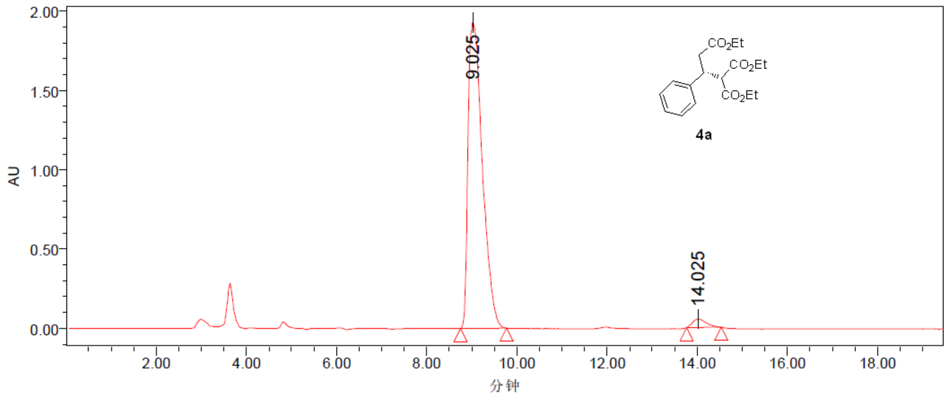
HPLC using an AD-H column (hexane: *i*-PrOH =90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	9.123	26964895	49.42	1472840
2	PDA 210.5 纳米	14.085	27594402	50.58	981586

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	9.123	26964895	1472840	49.42
2	PDA 210.5 nm	14.085	27594402	8981586	50.58

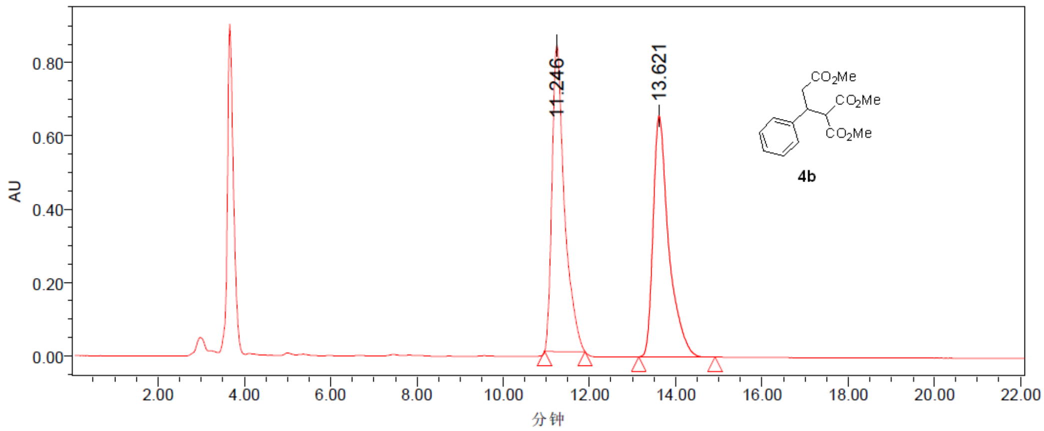


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	9.025	42603423	97.42	1927044
2	PDA 210.5 纳米	14.025	1129778	2.58	53512

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	9.025	42603423	1927044	97.42
2	PDA 210.5 nm	14.025	1129778	53512	2.58

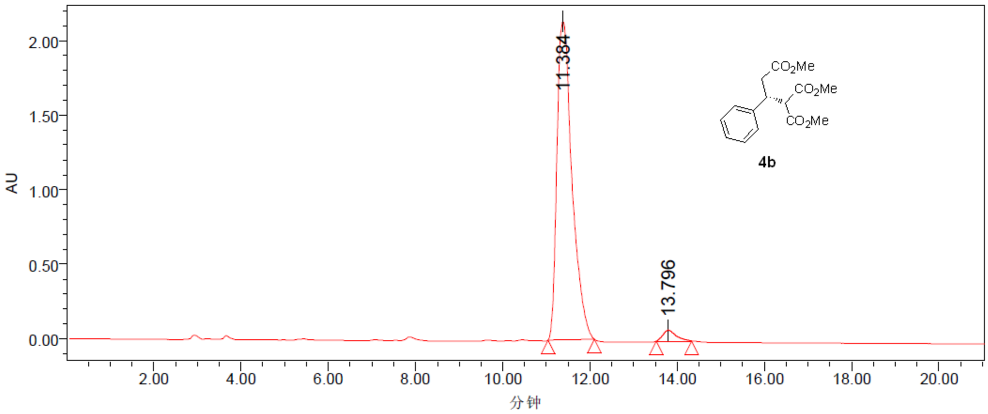
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 211.0** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 211.0 纳米	11.246	16814994	50.66	836394
2	PDA 211.0 纳米	13.621	16377680	49.34	657042

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 211.0 nm	11.246	16814994	836394	50.66
2	PDA 211.0 nm	13.621	16377680	657042	49.34

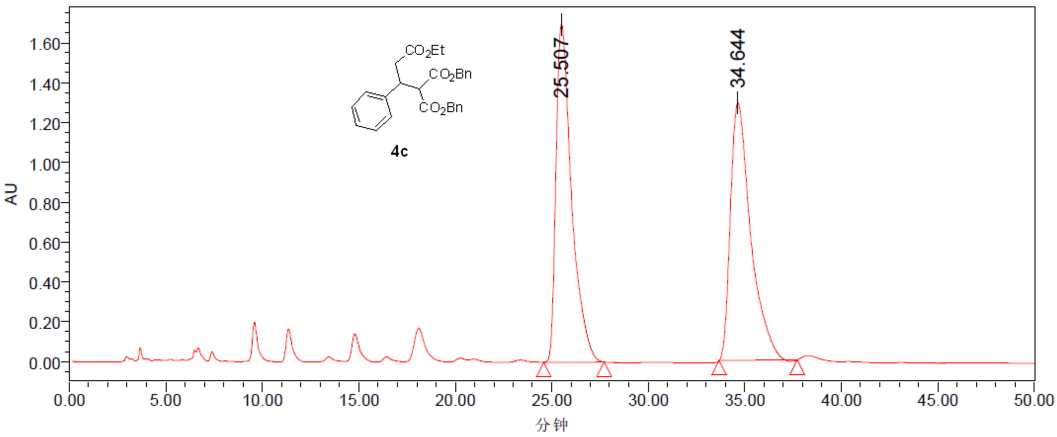


处理通道: **PDA 211.0** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 211.0 纳米	11.384	51425184	97.09	2137580
2	PDA 211.0 纳米	13.796	1541059	2.91	72643

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 211.0 nm	11.384	51425184	2137580	97.09
2	PDA 211.0 nm	13.796	1541059	72643	2.91

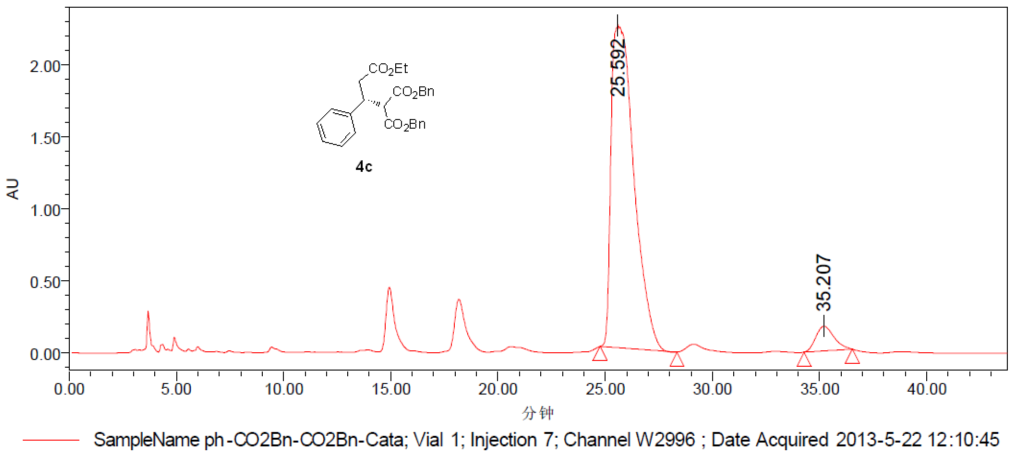
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	25.507	95618471	50.24	1695952
2	PDA 210.5 纳米	34.644	94722765	49.76	1292743

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	25.507	95618471	1695952	50.24
2	PDA 210.5 nm	34.644	94722765	1292743	49.76

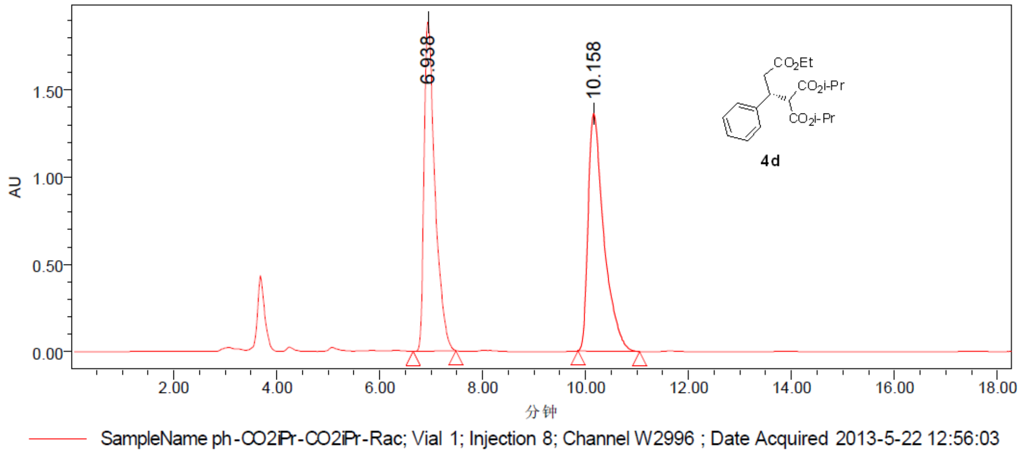


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	25.592	166312635	94.25	2238503
2	PDA 210.5 纳米	35.207	10143838	5.75	171027

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	25.592	166312635	2238503	94.25
2	PDA 210.5 nm	35.207	10143838	171027	5.75

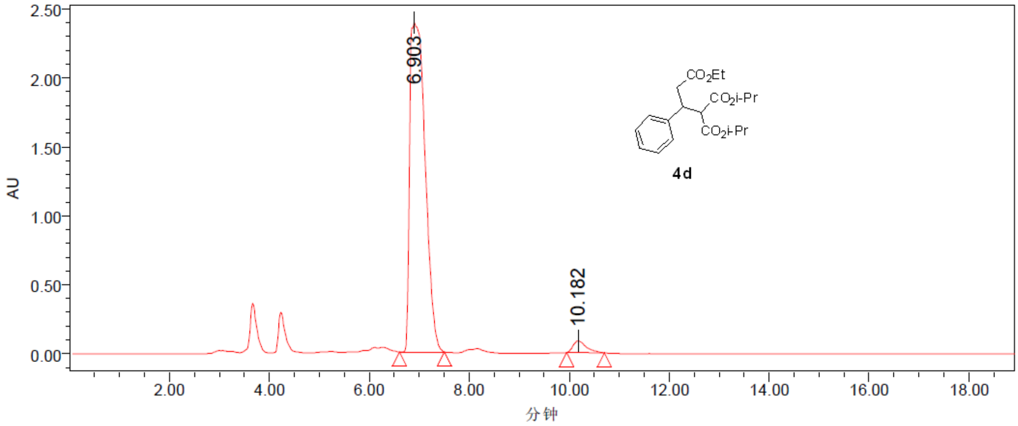
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	6.938	27905817	49.04	1888307
2	PDA 210.5 纳米	10.158	28994819	50.96	1363908

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	6.938	27905817	1888307	49.04
2	PDA 210.5 nm	10.158	28994819	1363908	50.96

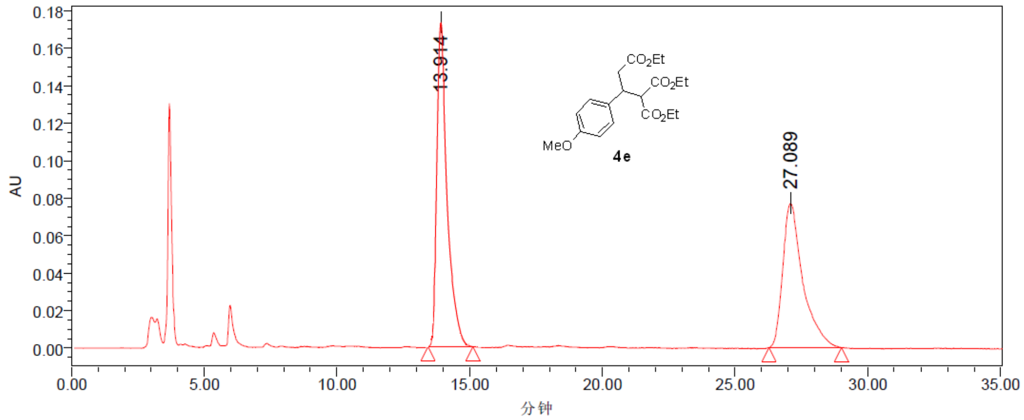


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	6.903	51296766	97.08	2386092
2	PDA 210.5 纳米	10.182	1545549	2.92	85530

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	6.903	51296766	2386092	97.08
2	PDA 210.5 nm	10.182	1545549	85530	2.92

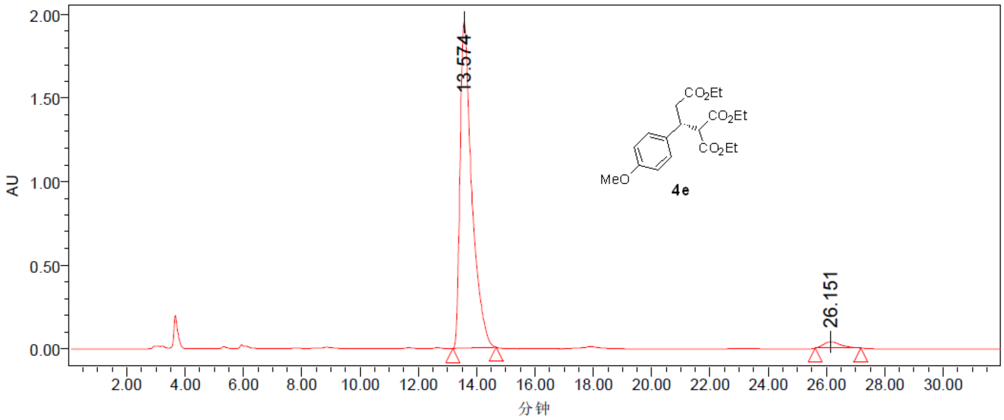
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	13.914	4160799	50.70	166814
2	PDA 210.5 纳米	27.089	4045664	49.30	76893

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	13.914	4160799	166814	50.70
2	PDA 210.5 nm	27.089	4045664	76893	49.30

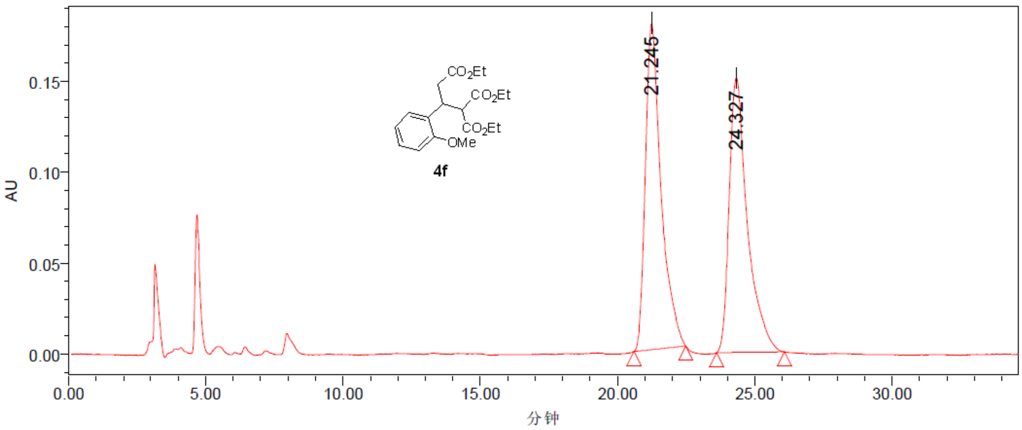


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	13.574	54643677	97.22	1950563
2	PDA 210.5 纳米	26.151	1561214	2.78	37462

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	13.574	54643677	1950563	97.22
2	PDA 210.5 nm	26.151	1561214	37462	2.78

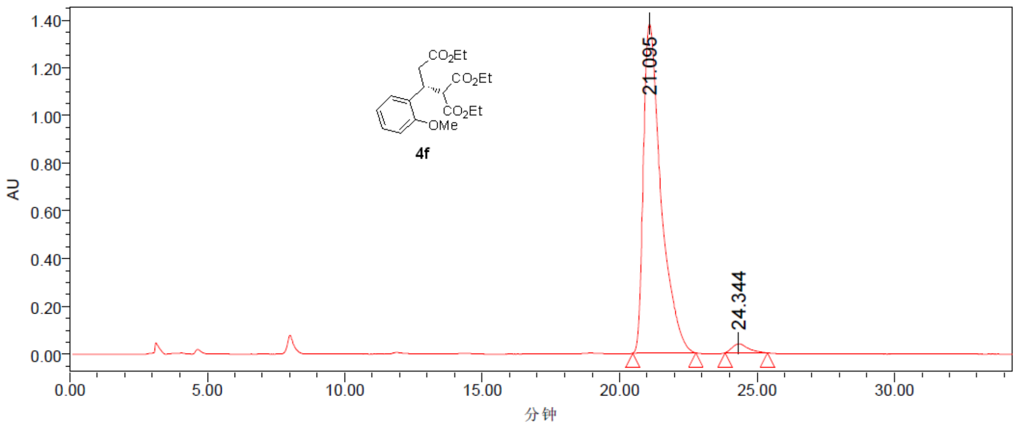
HPLC using an AD-H column (hexane: *i*-PrOH = 97:3, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	21.245	7124519	50.60	179355
2	PDA 210.5 纳米	24.327	6955588	49.40	150882

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	21.245	7124519	179355	50.60
2	PDA 210.5 nm	24.327	6955588	150882	49.40

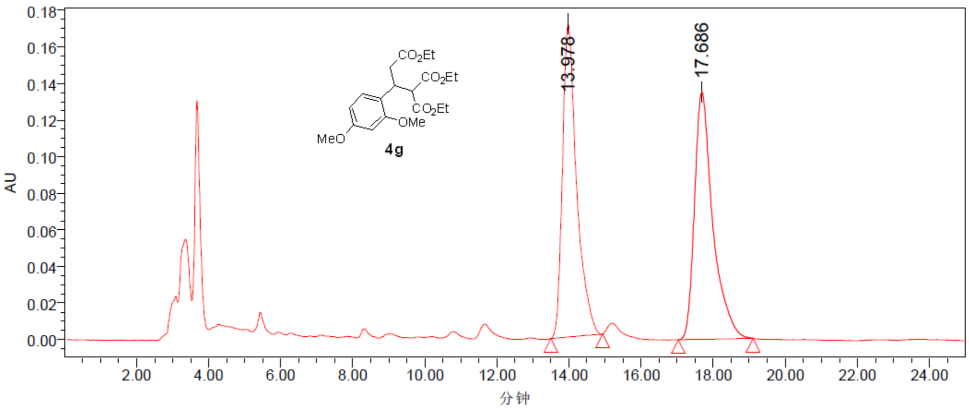


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	21.095	61210091	97.56	1379348
2	PDA 210.5 纳米	24.344	1529250	2.44	37835

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	21.095	61210091	1379348	97.56
2	PDA 210.5 nm	24.344	1529250	37835	2.44

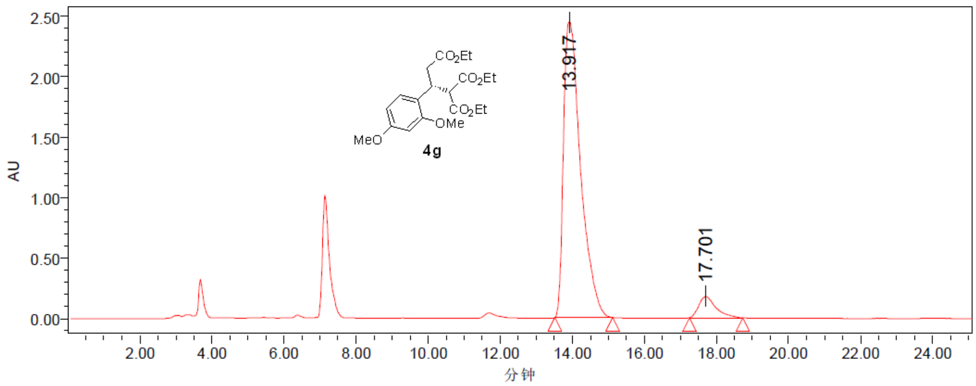
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	13.978	4663945	49.39	171086
2	PDA 210.5 纳米	17.686	4779541	50.61	135257

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	13.978	4663945	171086	49.39
2	PDA 210.5 nm	17.686	4779541	135257	50.61

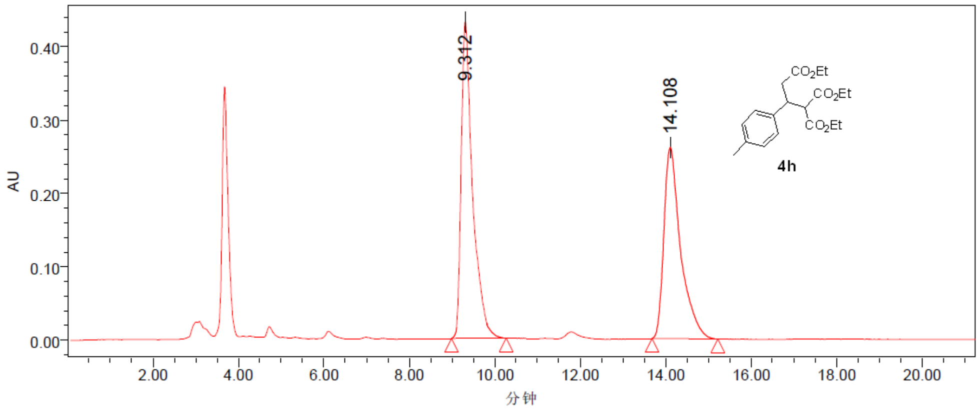


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	13.917	80641639	93.25	2444156
2	PDA 210.5 纳米	17.701	5838511	6.75	175843

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	13.917	80641639	2444156	93.25
2	PDA 210.5 nm	17.701	5838511	175843	6.75

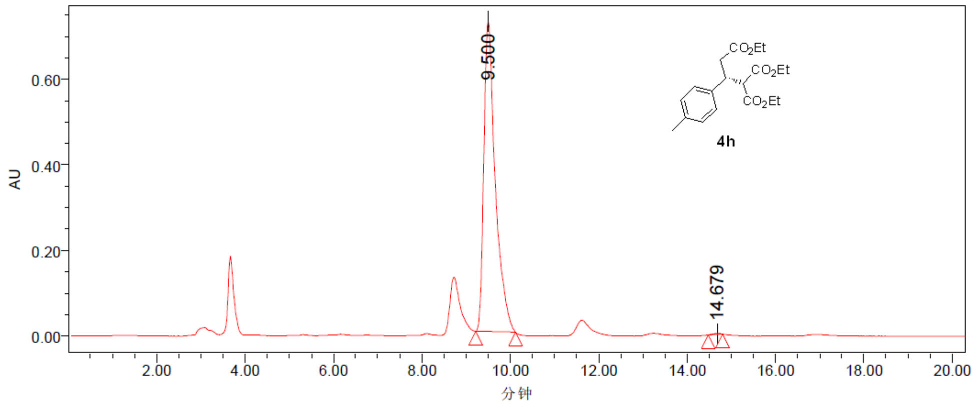
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.4** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.4 纳米	9.312	7316951	50.88	422955
2	PDA 210.4 纳米	14.108	7064737	49.12	260798

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.4 nm	9.312	7316951	422955	50.88
2	PDA 210.4 nm	14.108	7064737	260798	49.12

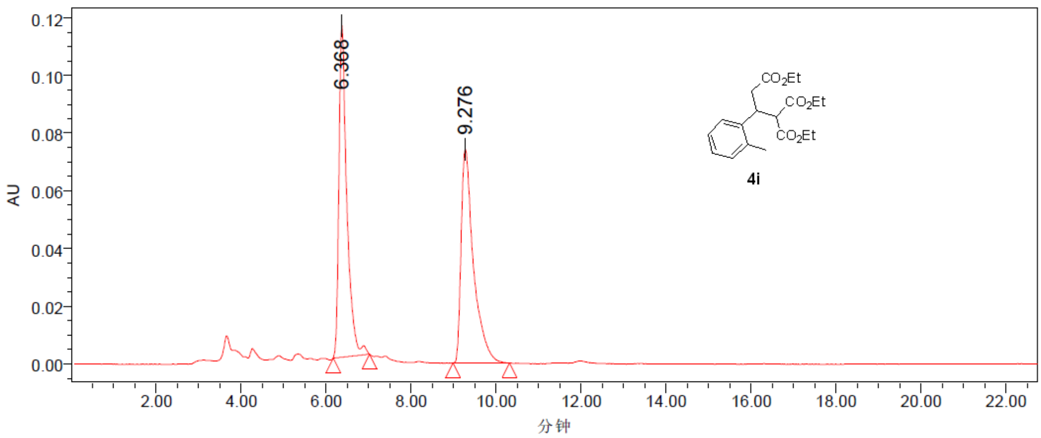


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	9.500	13045534	99.86	724603
2	PDA 210.5 纳米	14.679	18849	0.14	1539

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.4 nm	9.500	13045534	724603	99.86
2	PDA 210.4 nm	14.679	18849	1539	0.14

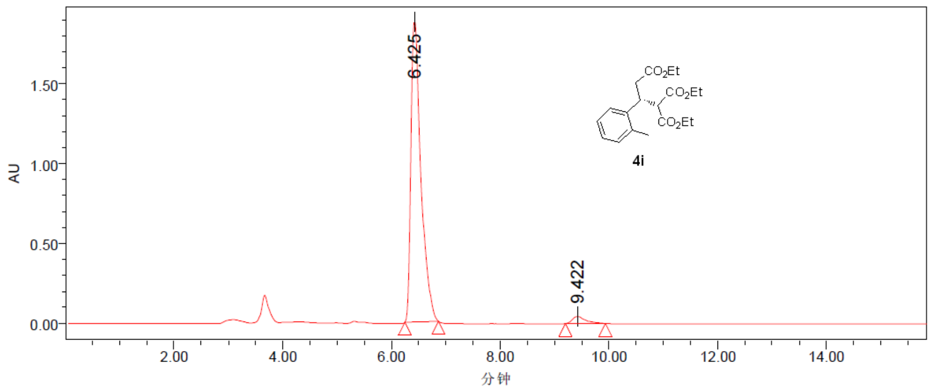
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: PDA 210.5 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	6.368	1506581	50.21	115162
2	PDA 210.5 纳米	9.276	1493741	49.79	74419

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	6.368	1506581	115162	50.21
2	PDA 210.5 nm	9.276	1493741	74419	49.79

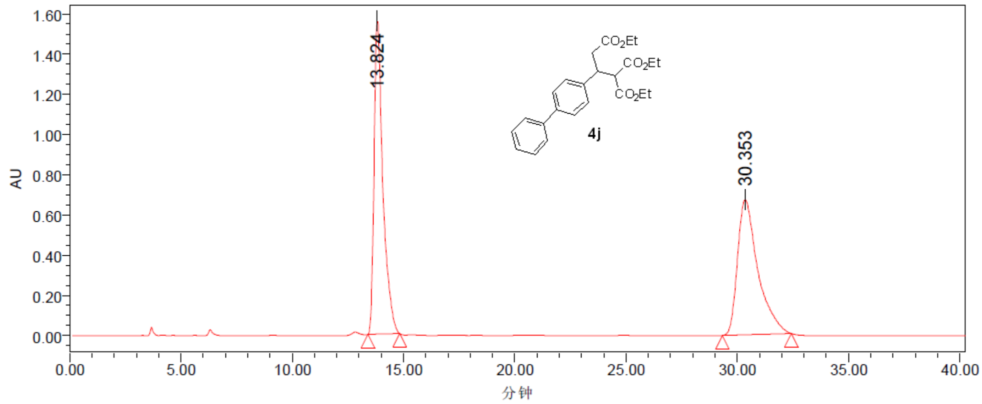


处理通道: PDA 210.5 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	6.425	24073411	97.00	1891622
2	PDA 210.5 纳米	9.422	744490	3.00	43938

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	6.425	24073411	1891622	97.00
2	PDA 210.5 nm	9.422	744490	43938	3.00

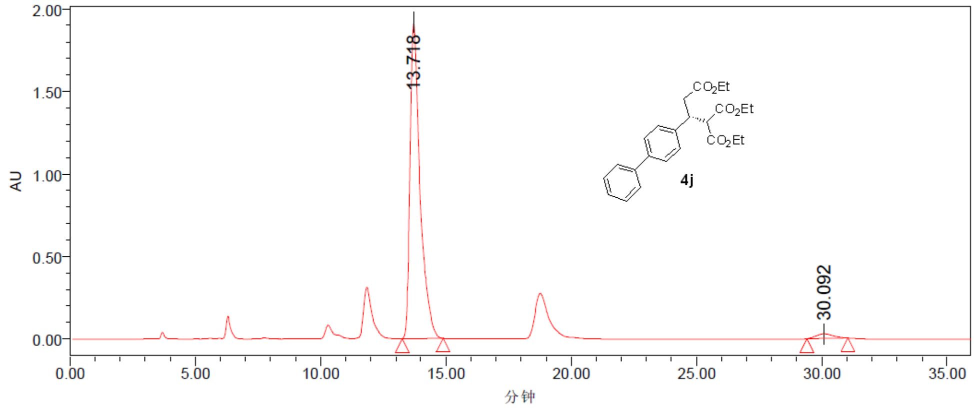
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 254.0** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 254.0 纳米	13.824	42890068	50.10	1559151
2	PDA 254.0 纳米	30.353	42724800	49.90	671554

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA254.0 nm	13.824	42890068	1559151	50.10
2	PDA 254.0 nm	30.353	42724800	671554	49.90

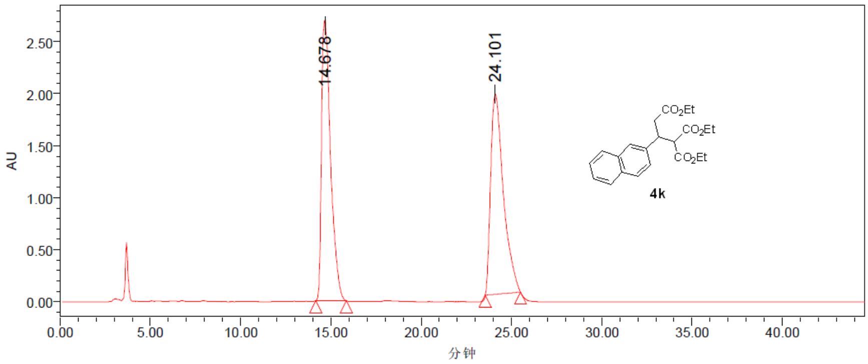


处理通道: **PDA 254.0** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 254.0 纳米	13.718	53233937	97.63	1916284
2	PDA 254.0 纳米	30.092	1294026	2.37	27110

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA254.0 nm	13.718	53233937	1916284	97.63
2	PDA 254.0 nm	30.092	1294026	27110	2.37

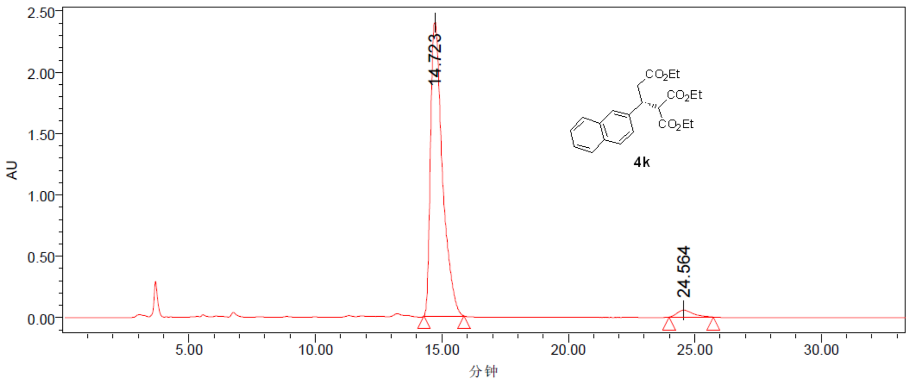
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	14.678	93771637	49.84	2671836
2	PDA 210.5 纳米	24.101	94364232	50.16	1925042

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	14.678	93771637	2671836	49.84
2	PDA 210.5 nm	24.101	94364232	1925042	50.16

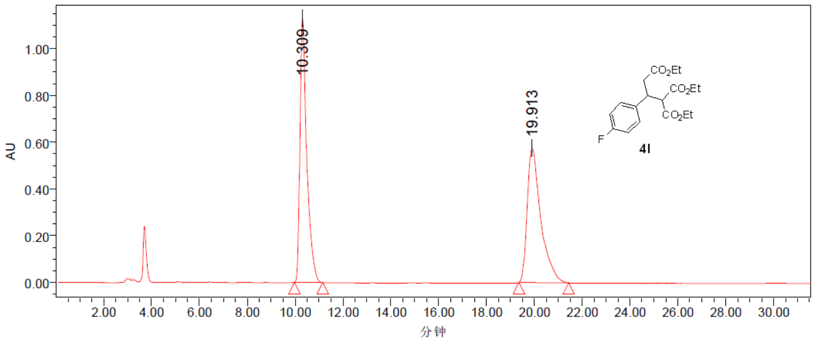


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	14.723	77666418	96.95	2401254
2	PDA 210.5 纳米	24.564	2440013	3.05	56372

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	14.723	77666418	2401254	96.95
2	PDA 210.5 nm	24.564	2440013	56372	56372

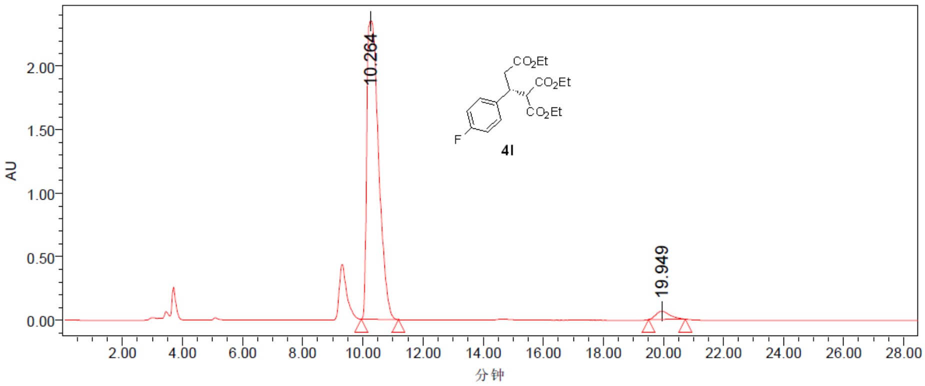
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	10.309	22575063	49.96	1132160
2	PDA 210.5 纳米	19.913	22612107	50.04	574265

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	10.309	22575063	1132160	49.96
2	PDA 210.5 nm	19.913	22612107	574265	50.04

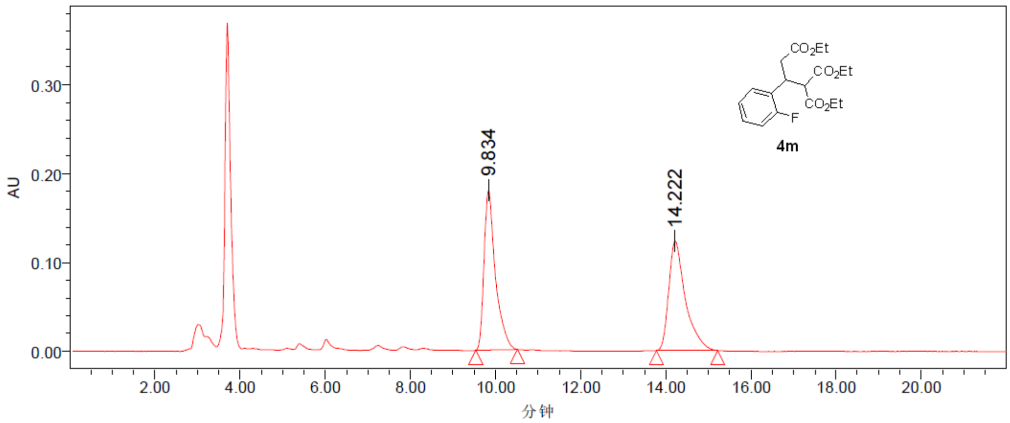


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	10.264	64395875	96.86	2352451
2	PDA 210.5 纳米	19.949	2090804	3.14	64542

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	10.264	64395875	2352451	96.86
2	PDA 210.5 nm	19.949	2090804	64542	3.14

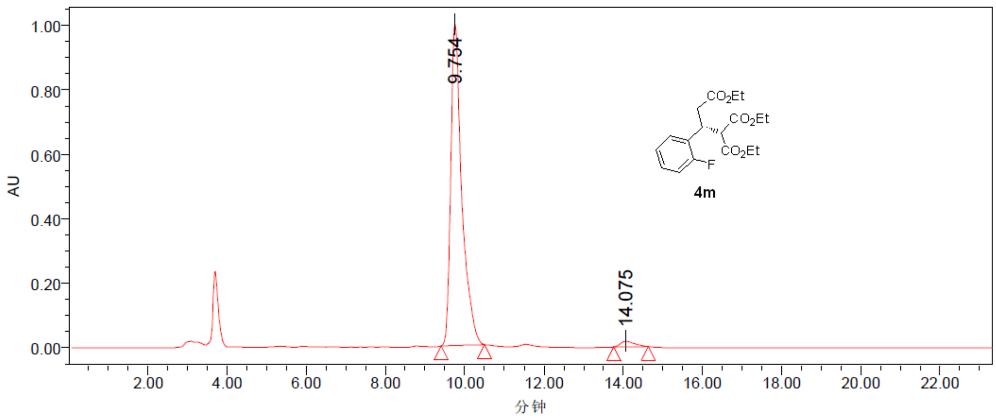
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	9.834	3322587	49.98	179548
2	PDA 210.5 纳米	14.222	3325065	50.02	123290

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	9.834	3322587	179548	49.98
2	PDA 210.5 nm	14.222	3325065	123290	50.02

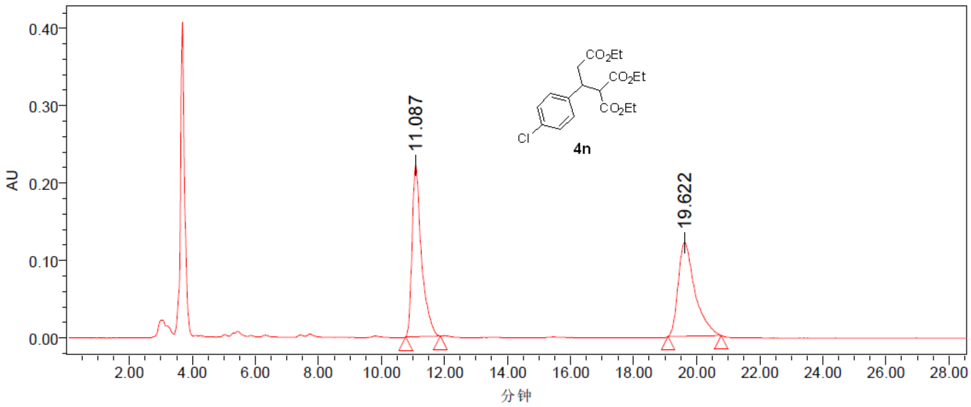


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	9.754	19388730	97.81	999563
2	PDA 210.5 纳米	14.075	434585	2.19	17599

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	9.754	19388730	999563	97.81
2	PDA 210.5 nm	14.075	434585	17599	2.19

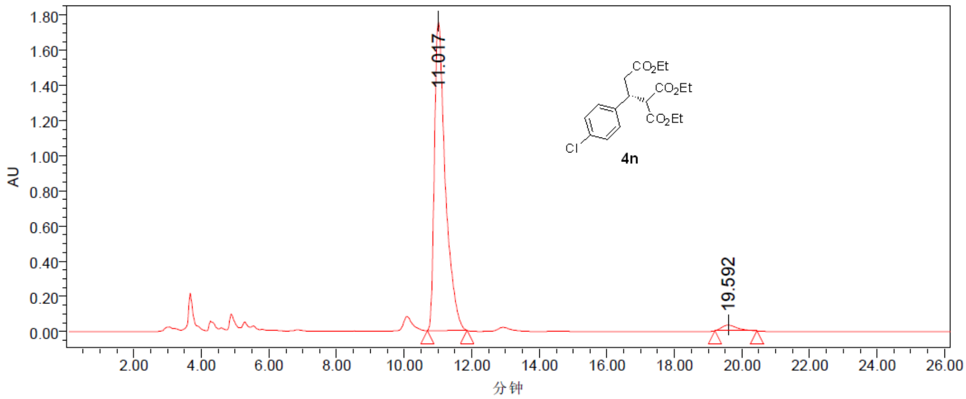
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	11.087	4596488	50.69	222252
2	PDA 210.5 纳米	19.622	4470699	49.31	121169

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	11.087	4596488	222252	50.69
2	PDA 210.5 nm	19.622	4470699	121169	49.31

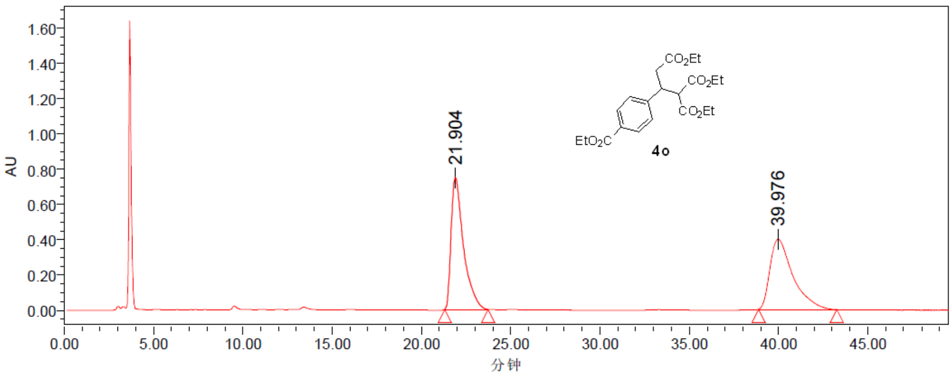


处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	11.017	39250010	97.39	1758523
2	PDA 210.5 纳米	19.592	1051600	2.61	32510

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA210.5 nm	11.017	39250010	1758523	97.39
2	PDA 210.5 nm	19.592	1051600	32510	2.61

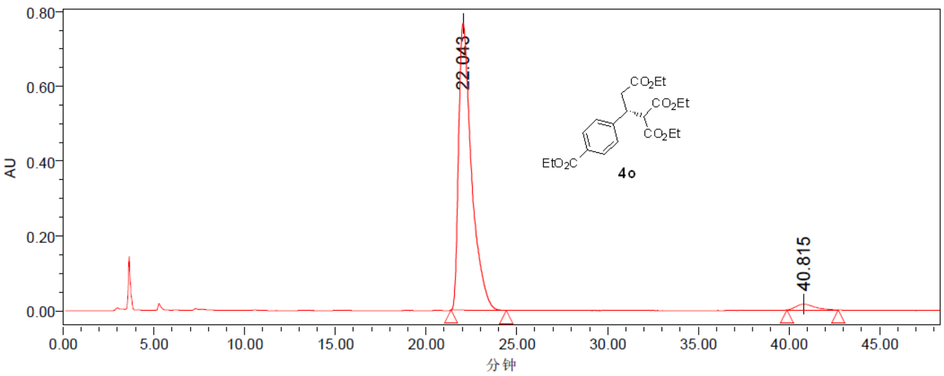
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 230.0** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 230.0 纳米	21.904	36030983	50.38	746918
2	PDA 230.0 纳米	39.976	35491256	49.62	397188

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 230.0 nm	21.904	36030983	746918	50.38
2	PDA 230.0 nm	39.976	35491256	397188	49.62

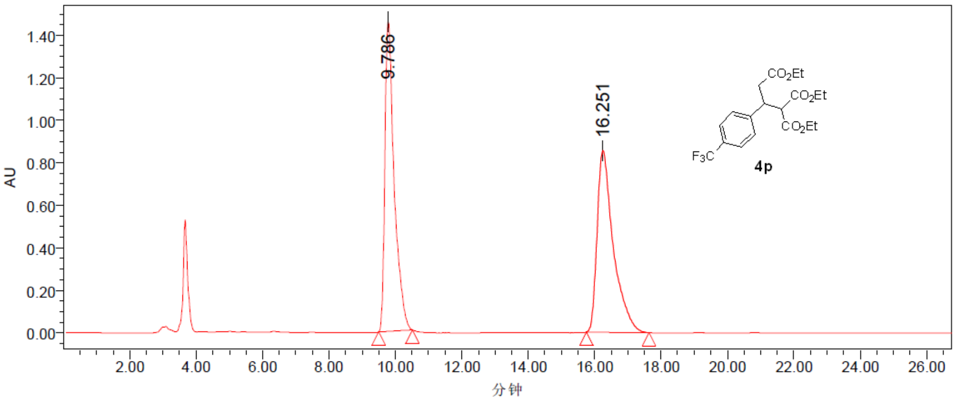


处理通道: **PDA 230.0** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 230.0 纳米	22.043	37895837	97.11	767337
2	PDA 230.0 纳米	40.815	1128918	2.89	15804

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 230.0 nm	22.043	37895837	767337	97.11
2	PDA 230.0 nm	40.815	1128918	15804	2.89

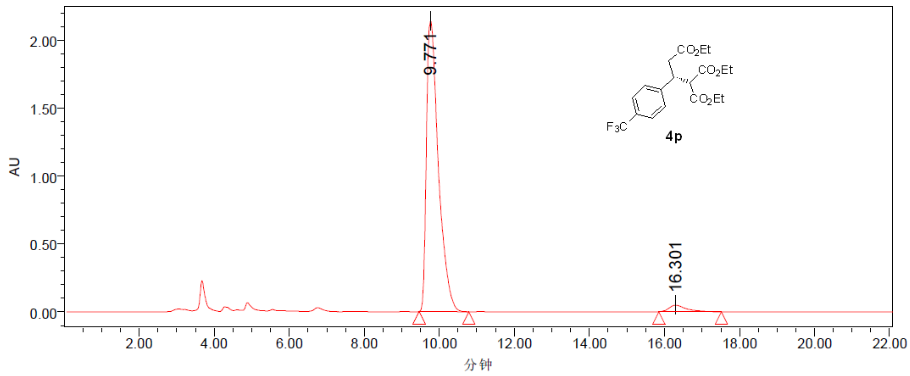
HPLC using an AD-H column (hexane: *i*-PrOH = 90:10, 1.0 mL/min)



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	9.786	28375290	49.48	1459942
2	PDA 210.5 纳米	16.251	28975178	50.52	855306

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	9.786	28375290	1459942	49.48
2	PDA 210.5 nm	16.251	28975178	855306	50.52



处理通道: **PDA 210.5** 纳米

	处理通道	保留时间 (分钟)	面积	% 面积	峰高
1	PDA 210.5 纳米	9.771	47118270	96.85	2135910
2	PDA 210.5 纳米	16.301	1533094	3.15	48042

Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA 210.5 nm	9.771	47118270	2135910	96.85
2	PDA 210.5 nm	16.301	1533094	48042	3.15